THE NICKEL BULLETIN

Monthly Abstracts of Recent Literature

JUNE

1960 Volume 33 No. 6

THE MOND NICKEL COMPANY LIMITED
THAMES HOUSE MILLBANK LONDON SW1
Telephone: VICtoria 9366

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NICKEL

Marketing of Nickel

K. H. J. CLARKE: 'The Marketing of Nickel.'

Canad. Mining and Metallurgical Bull., 1960, vol. 53, Feb., pp. 99–106.

The author discusses the marketing of nickel in sections concerned with the following aspects of the subject: sources of nickel, distribution, utilization, market development, and future developments. The review, much of which is based upon information relating to The International Nickel Company, Inc. (the main free-world producer), includes within its scope the principal free-world sources and suppliers of nickel.

The first section, *sources*, is concerned with the location of, and the activities centred upon, the three main types of nickel-ore deposit (sulphide ores, nickel-silicate ores, nickeliferous-iron ores), and with the production of nickel in Canada, New Caledonia, Cuba, the United States and elsewhere. Data supplementing the author's textual comments illustrate the increase in the free-world production of nickel from that in 1959 to the capacity planned for 1961.

In the section on *distribution*, the subject is discussed in terms of (*a*) North America, the largest geographical market for nickel, and (*b*) the other countries of the free-world: graphs illustrate the estimated consumption and deliveries of nickel in all forms for the years 1951–57.

The *utilization* of nickel is considered within the compass of a survey of the findings which, since Cronstedt first identified the metal in 1751, have led to the recognition of the industrial advantages offered by, and to the demand for, nickel.

An outline of the *markets* at present found by nickel (the various classes of nickel-containing material are correlated with their principal applications) forms an introduction to the author's comments on the *future developments* which are expected to widen the markets and meet the challenge presented by the increase in the free-world nickel-production capacity to an estimated 650,000,000 lbs. in 1961. Development work, it is anticipated, will be centred mainly on expanding the use of nickel in the following fields: gas-turbines for automotive applications, conventional automotive applications, plating, bridge construction, aeronautical structural applications, steel plants, nuclear engines, household products, architectural applications, mining, and the construction of railway carriages.

Decomposition of Formic Acid on Nickel Catalysts

J. FAHRENFORT, L. L. VAN REIJEN and W. M. H. SACHTLER: 'The Decomposition of Formic Acid on Metal Catalysts.'

Zeitsch. Elektrochemie, 1960, vol. 64, Apr., pp. 216–24.

During decomposition of formic acid on Ni/SiO_2 catalysts a considerable amount of formate is present on the surface, a fact which was demonstrated by means of I.R. spectroscopic techniques and by calorimetric determination of heats of adsorption. Mass-spectroscopic analysis of the gases remaining after adsorption of formic-acid vapour revealed the formation of a monomolecular layer approximating to the composition $Ni(OOCH)_2$. That the formate participated in the catalytic reaction was demonstrated by comparison of spectrokinetic I.R. data for adsorption or desorption with the kinetic data for the total catalytic reaction. The rate and activation energy of desorption of a completely covered surface were found to be comparable to the corresponding values for the catalytic reaction.

The intermediate formation of formate involves the discharge of hydrogen from the HCOOH molecule in two separate stages, an effect which is in agreement with the absence of CH_4 in the primary products resulting from catalytic decomposition of acetic acid and methyl formate.

The products formed during the decomposition of HCOOH through formate are closely analogous to those associated with pyrolysis of nickel formate. The results of mass-spectroscopic analysis show that H_2, CO_2, H_2O and CO occur as primary products during both reactions. In both cases the CO/CO_2 ratio is considerably higher than that corresponding to the water-gas equilibrium.

Since decomposition of HCOOH on other metal catalysts is also most probably governed by the properties of the corresponding formates, catalytic activities were compared with the stability of the relevant formates. The experimental data obtained demonstrate that the highest catalytic activity was exhibited by a series of metals which form formates in a relatively narrow range of stability.

Electrodeposition of Nickel Sheet

See abstract on p. 139.

Brazing of Nickel

See abstract on p. 157.

Check-Analysis Limits for Wrought Nickel

See abstract on p. 151

ELECTRODEPOSITION AND OTHER COATING METHODS

Restoring Confidence in Nickel/Chromium Plating

D. J. FISHLOCK: 'Restoring Confidence in Chromium Plating.'*

Metalworking Production, 1960, vol. 104, Apr. 6, pp. 601–7.

In collaboration with The British Non-Ferrous Research Association and The British Standards Institution, The Mond Nickel Company, Ltd., has launched a 'Quality Label' scheme which 'has for its objective the elimination of sub-standard electroplating'. The scheme was initiated in the light of the availability of an acceptable inspection method, an acceptable criterion of quality, and adequate supplies of nickel. In this article an attempt has been made to place the scheme within the general context of present-day plating developments and to outline its salient features.

The following are some of the points raised and discussed in the form of question and answer:

Has chromium plating deteriorated in quality in recent years?

It is considered unlikely that chromium plating has deteriorated in quality, particularly when 'quality' is assessed in the light of the increase in atmospheric polution and the greater complexity of the parts to be plated.

What about the cost of thicker deposits?

In terms of metal used, the cost of doubling the thickness of the nickel coating would be negligible. Prolonging the plating operation would add to the cost, but an increase in current density, temperature and agitation would usually ensure deposition of the thicker deposits with no increase in the operating time.

How can the thickness of a nickel deposit be measured quickly, accurately and non-destructively?

Thicknesses of nickel in the range covered by British Standard 1224 : 1959 can be determined with an accuracy of $\pm 0{\cdot}0001$ in. ($\pm 0{\cdot}0025$ mm.) by means of the B.N.F. thermo-electric plating gauge. The basic features of the gauge are outlined by the author (in this connexion, see, for further details, abstract in *Nickel Bulletin*, 1959, vol. 32, No. 12, pp. 351–2), but it is noted that the gauge is not suitable for measuring the thickness of the very thin chromium overcoatings conventionally used.

What is the recommended criterion of quality?

The main requirements of British Standard 1224: 1959 (the standard on which the label scheme is based) are detailed: see abstract and table in *Nickel Bulletin*, 1959, vol. 32, No. 12, pp. 350–1.

What long-term benefits do the organisers hope to achieve by the Quality Label scheme?

The propaganda campaign has the dual objective of increasing public confidence in the nickel/chromium finish, and of making the customer 'quality-conscious'. The labels, issued by the Company to manufacturers at cost price and in a variety of forms (e.g., adhesive, tie-on, printed tape), indicate by their colour whether a deposit is suitable for severe (red), moderate (blue), or mild (green) service. Prospective users are required to sign an undertaking that the labels will be employed correctly on plating *tested* to B.S. 1224: 1959.

What has been the reaction to the Quality Label scheme?

The scheme appears to be generally regarded as a 'good thing' and long overdue, and The Mond Nickel Company, Ltd., anticipate that the first labels will appear in the shops before the end of the year.

What notable advances have occurred in the practice of nickel/chromium plating to justify an opinion that its inherent quality may be improving?

Improvements in plating equipment and solutions, and the advantages of duplex deposits of nickel, are discussed. The improvements in corrosion-resistance associated with the use of duplex chromium and thicker and crack-free chromium overcoatings are reviewed, the rôle of the copper undercoat is briefly evaluated, and attention is drawn to suggestions that the presence of sulphur is deleterious in nickel deposits.

The user can, it is suggested, prolong the life of chromium plating by regular cleaning.

Throwing Power of Nickel and Other Plating Solutions

S. A. WATSON: 'The Throwing Power of Nickel and Other Plating Solutions.'

Trans. Inst. Metal Finishing, 1960, vol. 37, Spring, pp. 28–39.

Presented at the Annual Conference of the Institute, Scarborough, April 1960.

In the work described the throwing power of the various plating solutions studied was determined using the open Hull cell: throwing power is expressed in terms of the thickness of the deposit (measured at various points across the cathode) and of primary-current distribution (determined on an electrically-conductive paper model of the cell). Full details of the apparatus and techniques used are included in the paper, and the advantages of the procedure (*vis-à-vis* the Haring-Blum method) are discussed.

The reproducibility of the technique is exemplified by the author in relation to data derived from tests on Watts solutions prepared at intervals over a

* *We shall be pleased to supply a free reprint of this paper.*

period of several months. Data on the throwing power of various nickel, copper, chromium, tin-nickel and cobalt-nickel plating solutions are reported and compared.

The results obtained show that the open Hull cell provides an accurate measurement of throwing power. General trends in the results are independent of the primary-current ratios selected, reproducibility is good and the method is satisfactorily sensitive. Practical conditions are simulated more closely than with the conventional method employing the Haring-Blum cell, and it is possible to measure, in a single experiment, throwing power for several primary-current ratios. The need for accurate measurement of plating thickness by microscopical examination of sections may, however, restrict its application to the laboratory.

One objective of the investigation described was to explore the feasibility of improving the throwing power of nickel-plating solutions. It is known that high throwing power is favoured by decrease in the efficiency of deposition as the current density is raised, by achievement of high conductivity in the solution, and by attainment of suitable polarization characteristics (a steeply-sloping polarization curve). In the investigation, consideration was given to all three factors.

The throwing power of citrate, fluoborate, all-chloride and high-speed nickel solutions showed a moderate improvement compared with that of the Watts solution. Attempts to improve throwing power by complexing the nickel ions with fluorides and amines were unfruitful.

Studies are described in which the variation in the throwing power of nickel solutions was determined as a function of current density and of additions of alkali or alkaline-earth salts and organic substances. The author relates data for Watts solutions containing saccharin, and for similar solutions containing various concentrations of coumarin and of thiourea, to the effects of the respective addition agents on cathode potential at the appropriate concentrations. The relationship between levelling power and throwing power is discussed, and the potential conditions necessary for simultaneous achievement of high values in both properties are defined.

Contribution of Nickel and Chromium to the Durability of Decorative Plating

C. H. SAMPLE: 'The Contribution of Nickel and of Chromium to the Durability of Decorative Plating.' *Plating*, 1960, vol. 47, Mar., pp. 297-302.

The stated aim of the paper is '(*a*) to review some observations of the corrosion behaviour of various decorative plating systems, and (*b*) to present some opinions as to the reasons for the behaviour differences noted. It is hoped that these observations and opinions will shed some light on the seemingly-confusing current situation about 'how much and what kind' of nickel and of chromium should be used to provide the degree of corrosion protection and lustre required; and also encourage further research efforts in those areas where a better understanding of the basic factors appears to be needed.'

In an introductory section, reference is made to work which has demonstrated that an increase in the thickness of the chromium constituent of nickel/chromium coatings is beneficial only when the undercoating is of bright nickel, and might, in the case of buffed dull-nickel undercoatings, even be deleterious. From the standpoint of corrosion-resistance, fully-bright nickel is generally inferior to a buffed dull-nickel coating of the same thickness when overplated with a conventional chromium deposit of a conventional thickness. Also raised is the question of whether a copper undercoating improves the corrosion-resistance of chromium/nickel coatings.

These general points are then particularized with respect to the advantages associated with duplex-nickel coatings (semi-bright nickel of high-levelling characteristics + fully-bright nickel + a conventional thickness of chromium), with bright-nickel + thicker-chromium coatings, and with copper + nickel + chromium (*vis-à-vis* nickel + chromium) coatings. His discussion of the findings reported in the literature leads the author to conclude that duplex-nickel coatings over-plated with conventional chromium of a conventional thickness confer corrosion-resistance substantially equivalent to that of chromium-plated buffed-dull-nickel coatings of the same thickness.

The application of thicker-than-normal chromium coatings to those bright-nickel deposits which are adversely affected by conventional chromium plating markedly improves the corrosion-resistance of the composite. Such coating systems are, however, relatively new, and further experience is considered necessary before any assessment can be made of their advantages, in terms of economy and long-term durability, over an improved nickel substrate.

Copper + bright-nickel + conventional-chromium coatings confer, for a limited time, better protection than comparable coatings without copper, but, at best, such coatings are considered to fall far short of the high standards of durability established long ago in the industry with buffed-dull-nickel + conventional-chromium coatings and now available with duplex-nickel systems.

One section of the paper is devoted to consideration of the reasons for variations in the corrosion behaviour of nickel/chromium coatings, and of the rôle of the final chromium layer. The following mechanism is advanced in explanation of the observed facts:

'Hard, exceedingly-fine-grained (and perhaps micro-stressed) nickel coatings are prone to fine cracking when they are chromium plated. It would appear that such cracking occurs during the very first moments of the operation. The mechanism of the cracking phenomenon, while at present obscure, is probably either some sort of stress-corrosion cracking, or so-called hydrogen cracking, or perhaps a combination of both.

'Due to the very fine nature of the cracks involved, together with the excellent micro-throwing power of chromium-plating solutions, such cracks and their intersections are, in effect, filled or bridged over with chromium when chromium plating is continued beyond the time required to apply conventional thicknesses (e.g., 0·02 mil or less). Thus, in effect, such coatings are substantially pore-free initially, and as a result do not exhibit rapid failure. With conventional chromium-plating thicknesses, however, only the very finest cracks apparently are bridged or sealed, but the wider cracks and intersections of two or more are not. Thus such coatings are not pore-free initially, and as a result exhibit early failures in environments such as at Kure Beach.'

Methods of Improving the Resistance of Nickel/Chromium Coatings to Atmospheric Corrosion

H. BROWN and D. R. MILLAGE: 'Methods of Obtaining Improved Outdoor Corrosion-Resistance with Nickel + Chromium Plate.'

Trans. Inst. Metal Finishing, 1960, vol. 37, Spring, pp. 21–7.

Presented at the Annual Conference of the Institute, Scarborough, April 1960.

Extensive studies to evaluate the corrosion-resistance of various combinations, thicknesses and types of copper, nickel and chromium electrodeposit are continuing in an attempt to develop plating procedures or composite coatings which will confer increased protection on zinc-base die castings and steel components exposed to the atmosphere during service. In this paper the authors outline the characteristic features of nickel/chromium coating systems which have been evolved with this aim in mind, and discuss, in the light of the corrosion data available, the advantages offered by each.

In the introductory section of the review the authors consider the factors involved in the mechanism underlying the corrosion of nickel/chromium electrodeposited coatings (under the corrosive conditions to which plated car components are exposed when large amounts of salt are used to de-ice the streets of industrial cities in winter). The corrosion-resistance of the following composite-coating systems is then considered in relation to the variables involved:—

(1) Bright crack-free chromium in thicknesses of 0·03–0·08 mil (0·75–2 micron) deposited over nickel or copper+nickel coatings.

(2) Conventional 0·01-mil-thick chromium deposited on a duplex-nickel coating (consisting predominantly of a deposit of soft semi-bright sulphur-free nickel+a thin overlay of bright nickel).

(3) A duplex microscopically-cracked bright chromium coating (0·03–0·1 mil thick) deposited on nickel or copper+nickel coatings.

(4) Chromium + nickel + chromium composite coatings.

Systems (2) and (3) are considered the best in use to date, and of these, system (2) is well established and in more widespread use. System (3) is deemed to offer considerable promise, though the relevant outdoor-exposure tests have not been in progress long enough to permit its proper assessment. System (4) has not been evaluated under production conditions, but the results of extended (4-year) outdoor-exposure tests indicate that it is capable of offering the highest overall corrosion-resistance.

Corrosion-Resistance of Chromium/Nickel-Plated Zinc-Alloy Die Castings: Influence of Chromium Thickness and Basis Metal

O. J. JONES, V. E. CARTER and J. EDWARDS: 'Factors Influencing the Corrosion-Resistance of Decorative Plating on Zinc-Alloy Die Castings.'

Trans. Inst. Metal Finishing, 1960, vol. 37, Spring, pp. 40–9.

The work described was carried out in two phases and with two aims in view: (1) to determine the influence, on the durability of plated zinc-alloy castings, of variations in the structure of the basis metal and of the presence of typical casting defects; (2) to investigate the feasibility of improving corrosion-resistance by use of thicker chromium coatings (deposited under modified conditions) and by substitution of nickel for the conventional copper undercoat. In both parts of the programme the opportunity was taken of studying the effects of varying the thickness of the main, bright-nickel coating.

Previous work had indicated that the principal mode of service failure in plated die castings is by penetration of the copper+nickel+chromium coating (under the influence of atmospheric corrodents), followed by attack on the zinc-alloy basis metal and subsequent formation of voluminous solid corrosion products which raise the coating around each point of penetration (see, for example, paper by CARTER and EDWARDS abstracted in *Nickel Bulletin*, 1958, vol. 31, No. 5, p. 130). Because of the generally poor performance of plated die castings, many platers have believed that the quality of the casting is an influential factor in this respect. To provide more conclusive evidence on this point, a programme of atmospheric-exposure tests was carried out on plated samples of zinc die-casting alloy fabricated in various ways (including conventional pressure die-casting, pressure die-casting in an evacuated mould, rolling from a cast ingot, and gravity die-casting): it was expected that these methods would produce a range of surface structures and would eliminate most of the surface defects typical of pressure die castings. Some of the pressure die castings and rolled sheet were plated in two industrial plating shops, but samples produced by all the methods mentioned were plated in the laboratory. The nature and position of surface defects on all of the industrially-plated specimens were recorded so that their influence on corrosion behaviour could be ascertained.

In the second phase of the programme, chromium was deposited, under 'crack-free' conditions, to a thickness greater than normal. Pressure die castings

were plated with copper (or nickel) + bright nickel +chromium. The influence of the bright nickel constituent of the composite coating was studied at three thickness levels, and that of the chromium at five levels of thickness up to $0{\cdot}2\times10^{-3}$ in.

Specimen preparation, plating procedures and test conditions are fully described.

From the data presented it is concluded that variations in surface structure, or the presence of minor defects, did not significantly influence the service performance of plated castings still commercially acceptable from the point of view of appearance. Accidental variations in the chromium thickness of the composite coating were much more influential.

The exposure tests, in which the previously reported benefits of thicker crack-free chromium were investigated, are still in progress, but results to date indicate a definite increase in resistance to blistering with increase in chromium thickness. The optimum minimum thickness of chromium may lie within the range $0{\cdot}05$–$0{\cdot}1\times10^{-3}$ in., but it appears that even a modest increase in thickness from the conventional $0{\cdot}01\times10^{-3}$ in. to about $0{\cdot}03\times10^{-3}$ in. would add significantly to corrosion-resistance. On all the types of specimen studied an increase in the thickness of the bright-nickel undercoating proved beneficial, and it is emphasized that although a thicker layer of chromium will confer added protection, there are no grounds for reducing the thickness of the nickel below the values laid down for the particular service conditions in the current British Standard. The atmospheric corrosion tests are being continued, so as to permit an accurate assessment of (1) the improved resistance to blistering conferred by the thicker chromium coating, and (2) the deleterious effects of the presence of cracks in the chromium.

Substitution of nickel for the conventional copper undercoat did not appear to increase corrosion-resistance, but the results of these tests are regarded of doubtful validity in the light of previous findings.

Heat-Treatment of Electrolessly-Deposited Nickel Coatings

K. T. ZIEHLKE, W. S. BRITT and C. H. MAHONEY: 'Heat-Treating Electroless Nickel Coatings.'

Metal Progress, 1960, vol. 77, Feb., pp. 84–7.

Although electrolessly-deposited nickel (a chemically-deposited nickel-phosphorus alloy) is known to be heat-treatable to high hardness, relatively little information is available in the literature on the relationship between hardness and heat-treatment cycle. This paper summarizes the results of an investigation carried out, at the Oak Ridge Gaseous Diffusion Plant, with the aim of studying the response of such deposits to heat-treatments covering a wide range of times and temperatures. The influence, in this connexion, of the phosphorus content of the deposits was also determined.

Mild-steel and low-alloy chromium-steel specimens were electrolessly nickel plated and then heat-treated. The variation in hardness of a coating containing 8·5 per cent. phosphorus is plotted in the paper as a function of heat-treatment at temperatures in the range 480°–1380°F. (250°–750°C.) for times ranging from 3 minutes to 3 hours. Response to very short-time heat-treatment was studied in tests on an 8·7 per cent. phosphorus coating.

Photomicrographs revealed no changes in the microstructure of samples heat-treated below 750°F. (400°C.), although hardness increased from the as-deposited value to a maximum of about 1000 D.P.H., the highest hardness resulting from the heat-treatments studied. At temperatures higher than 750°F. precipitation and agglomeration were detected: agglomeration became pronounced with increase in temperature and time of heat-treatment and was more rapid in coatings of low phosphorus content. After heat-treatment for 1 hour at 750°F. to obtain maximum hardness, the laminated structure of the as-deposited coating was still prominent, but at 930°F. (500°C.) the laminations were less conspicuous and a precipitate had formed. Agglomeration of the precipitate had commenced at 1110°F. (600°C.) and hardness had fallen to 725 D.P.H. Resolvable particles were present in the microstructure after heat-treatment at 1380°F. (750°C.).

The increase to maximum hardness in the first stages of heat-treatment is considered attributable to the establishment of an interim crystal lattice in the initially amorphous deposit. Since no diffusion is required, only a small amount of atom movement is sufficient to form the lattice from the amorphous structure, and at high temperatures the transformation is rapid. (The transformation is exothermic, indicating the establishment of an ordered structure.) The high hardness of this new lattice arrangement is probably the result of distortion caused by the non-equilibrium locations of the atoms of nickel and phosphorus. As heat-treatment continues, atoms diffuse to more stable locations, with subsequent decrease in hardness.

The results indicate that phosphorus does not play an important rôle in the initial response of electroless nickel to heat-treatment; after maximum hardness has been reached, however, the phosphorus content has a pronounced effect on the degree to which the deposit softens. Softening during prolonged heat-treatment was dependent on co-precipitation and agglomeration of nickel and nickel phosphide from the pseudostable interim lattice. The hardness of the coatings after prolonged heat-treatment appeared to be a function of the amount of Ni_3P present. At a given level of hardness, ductility was found to decrease with increase in the phosphorus content.

Age Hardening of Electrolessly-Deposited Nickel Coatings

W. G. LEE: 'Age Hardening of Chemical Nickel Coatings.'

Plating, 1960, vol. 47, Mar., pp. 288–90.

The work reported forms a continuation of a previous investigation of the influence, on the hardness of chemically-deposited nickel, of annealing for

various times at various temperatures. GOLDSTEIN *et al.* had studied the change in the structure and hardness of such deposits as a function of exposure in an inert atmosphere at temperatures of 200°, 300° and 400°C. (see *Jnl. Electrochemical Soc.*, 1957, vol. 104, Feb., pp. 104–10: abstract in *Nickel Bulletin*, 1957, vol. 30, No. 6, p. 89). In the present investigation, variation in hardness of electrolessly-deposited nickel coatings was studied during exposure for various times at 190°, 232°, 260°, 288° and 315°C. The coatings were deposited from a 'Kanigen' plating solution, and contained 8–10 per cent. of phosphorus. In thickness they ranged from 3–5 mils. Since it was hoped that low-temperature annealing (200°–300°C.) would improve mechanical properties without the formation of an oxide film deleterious to the deposit's appearance, the coatings were annealed without the protection of an inert atmosphere.

The results show that the time required to reach maximum hardness decreased with rise in the annealing temperature. At 200°C. no change in hardness was observed after a value of 600 V.H.N. was attained as a result of annealing for 617 hours. At 232°C. maximum hardness (925 V.H.N.) occurred after 40 hours, but the curve for the 260°C. isotherm appears abnormal (the maximum hardness attained was 850 V.H.N.), and some doubt is cast on the data obtained. Heat-treatment at 288°C. resulted in the attainment of maximum hardness somewhere in the range 2–18½ hours; oxidation was, however, observed after 23 hours of annealing.

From a practical point of view, exposure for 15–20 hours at about 280°C. (in an ordinary furnace without use of a protective atmosphere) is considered capable of conferring maximum hardness and wear-resistance. For maximum impact-resistance, on the other hand, recourse to a lower temperature (240°–260°C.) and a longer annealing period is recommended.

Use of Disodium M-Benzenedisulphonate as a Hardening Agent in Nickel Plating

W. H. METZGER, P. A. KRASLEY and F. OGBURN: 'Use of Disodium M-Benzenedisulfonate as a Hardening Agent in a Watts Nickel Bath.'

Plating, 1960, vol. 47, Mar., pp. 285–7.

With the introduction of high-speed rotary presses at the U.S. Bureau of Engraving and Printing, the need arose for a relatively thin all-nickel electroformed intaglio printing plate. The electroforms produced using a Watts-type solution were subjected to as much as 0·1 per cent. elongation after repeated printing, and it was thought that a harder nickel deposit would eliminate this undesirable characteristic. A reduction in the internal stress of the electroform was also deemed advantageous. The subsequent research, described in this paper, culminated in the recommendation that disodium m-benzenedisulphonate should be used as an addition agent to the Watts solution, which was to be operated under the same conditions as hitherto (5 amp./dm.², 50°C., pH 5·0).

Electrodeposits produced in the presence of such additions fully satisfied requirements. The hardness of the coatings increased to a maximum of 350 V.H.N. at a concentration of 0·3 g./L. As the amount of additive present was increased from 0 to 0·4 g./L. the stress varied from 20,000 p.s.i. (9 t.s.i.; 14 kg./mm.²) tensile to 5,000 p.s.i. (2 t.s.i.; 3·5 kg./mm.²) compressive. The additions caused hardness to vary inversely with pH within the range studied (pH 4–5). The relative stability of the agent appeared to be unaffected by conventional purification procedures.

A study of the data obtained using two contractometers fitted with coils differing in age and condition demonstrated that the discrepancy in the stress values obtained was attributable to the surface condition of the coils, the 'stop off' procedure used on the inside of the coil, and the mechanical differences between individual contractometers.

Protection of Uranium by Electrodeposition and Diffusion of Nickel

C. CHAUVIN, H. CORIOU and J. HURE: 'Protection of Uranium by Electrodeposition and Diffusion of Nickel.'

Electrochimica Acta, 1959, vol. 1, July, pp. 177–89.

Uranium oxidizes rapidly in air and in aqueous environments, and in nuclear-reactor applications the engineer is therefore confronted with the problem of conferring on the uranium fuel element the corrosion-resistance necessary to eliminate the dangers (e.g., introduction of fission products into the heavy-water coolant) otherwise involved in any rupture of the canning material. In the case of aluminium canning, the problem is complicated by the need to provide a diffusion barrier between the uranium element and the aluminium. To provide the requisite protection a metallic coating would ideally possess the following characteristics: a small neutron-capture cross section; freedom from porosity; satisfactory resistance to corrosion by water at 100°C.; the capacity to produce a diffusion bond with, and thus adhere firmly to, the uranium (the diffusion zones being sufficiently ductile to withstand the subsequent canning operations). The investigation now described was initiated to determine the suitability in this respect of electrodeposited nickel. The uranium-nickel equilibrium diagram indicates the existence of seven intermetallic compounds, but, although the use of nickel might be objected to on this account, the degree to which the metal otherwise satisfies the requirements renders it of particular interest.

The investigation involved the following phases:

(1) *Nickel Plating.* The initial experiments were carried out using a Watts plating solution, and the adherence of the electrodeposited nickel coatings to uranium was studied as a function of surface treatment (anodic and chemical cleaning). Chemical cleaning achieved reproducible and satisfactory results and was employed in the remainder of the study.

The 20μ-thick nickel deposits obtained from the Watts solution (BO_3H_3 37 g./L.) used in the initial studies of surface treatment contained a prohibitive amount of boron (300–500 p.p.m.). It was therefore necessary to employ a plating solution free from boric acid, and a solution of the following composition was found suitable, both in relation to the adherence of the deposit immediately after nickel plating and the quality of the diffusion zone after subsequent treatment:

	g./L.
$NiSO_4.7H_2O$	70·4 (0·5N)
NH_4Cl	5·4 (0·1N)

Plating conditions studied:

temperature	25–40°C.
pH	3·5–6
current density	1–3 amp./dm.²

(2) *Diffusion Treatment.* Diffusion studies were carried out in two stages: one involved prolonged pre-diffusion (or degassing) at a low temperature; in the other, which comprised the main diffusion treatment, the nickel-plated uranium was heated *in vacuo* at 600°, 700° or 800°C. Heating and cooling rates did not exceed 100C.°/hour. Photomicrographs supplement the data presented on the influence of the following variables on the resultant diffusion: diffusion temperature; the type of plating solution; the purity of the uranium; the thickness of the nickel deposit. The procedures used in preparing the specimens for metallographic examination are described.

The authors draw attention to the following points, which are considered of importance in relation to the possible use of nickel as an intermediate layer:

1. After suitable chemical pre-treatment, uranium can be plated with nickel coatings of an adherence ensuring satisfactory diffusion during subsequent annealing without application of pressure. This diffusion zone reinforces the corrosion-resistance conferred on the uranium by the aluminium canning.

2. Other conditions being equal, the quality of the diffusion zones obtained depends on the type of nickel-plating solution employed.

3. Microscopical examination of the diffusion zone after annealing for 100 hours at 700°C. revealed the existence of five layers (six were determined using polarized light), four of which were very crystallized.

4. The significant irregularities found at the interface between the uranium and the first intermetallic compound (U_6Ni) appeared to be due to the presence of certain impurities in the uranium which acted as diffusion barriers.

5. Of the seven compounds capable of forming during diffusion, the hardest and most brittle would be the richest in nickel: UNi_5. This brittleness was reflected in fractures at the Ni/UNi_5 interface.

Electrodeposition of Nickel and Nickel/Copper Bi-Metal Sheet

E. CARRINGTON: 'The Electrodeposition of Copper, Nickel and Bi-Metal Sheets.'

Electroplating and Metal Finishing, 1960, vol. 13, Mar., pp. 80–4.; Apr., pp. 126–9, 143.

Whilst it is relatively easy to roll an ingot of metal down to sheet of moderate thickness, it is virtually impossible to roll large sheets down to a thickness of a few mils and yet maintain an overall uniformity of thickness. The thinner the sheet required, the higher the rolling costs. Electrodeposition, however, offers a method of forming sheet, the cost of which decreases with decrease in the thickness required. In this article the author outlines experience gained in the electroforming, on a production scale, of copper, nickel and copper/nickel sheet. The limiting thickness (i.e., that at which the costs of rolling and electroforming coincide) was found to be in the region of 21 gauge (35 mil). It was decided therefore to restrict the application of the electroforming technique to the production of sheet 12×4 ft. (3·5×1·2 m.) in area and less than 35 mil thick. The copper sheet was to be used for motor-car gaskets and radiators, the nickel for radio-valve parts, and the slightly thicker bi-metal sheet (consisting of 23 mil of copper and 5 mil of nickel) for the manufacture of saucepans.

Requirements necessary for economic deposition of such sheet are noted, operating conditions are outlined, and plant layout is described. Other sections cover the following subjects: process control, bi-metal deposition, filtration problems and those encountered in the production of copper sheet, anodes used, copper recovery, water consumption and electrical plant. The article includes photomicrographs illustrating structures typical of electrodeposited sheet.

The sheets were electroformed on revolving stainless-steel mandrels. The copper solution was operated at 200 amp./sq. ft. (21·5 amp./dm.²) and 40°–50°C. The composition of the nickel solution, which was operated at 102 amp./sq. ft. (11 amp./dm.²) and 75°C., is noted below:

	g./L.
nickel sulphate	280
boric acid	30
potassium chloride ..	2–10

Sulphuric acid was added, as required, to maintain the pH at 3·2–3·8. The nickel-sulphate concentration was not critical, but care was taken to ensure that saturation point was not approached. Boric acid was used as a buffer, the saturation point being almost reached at a concentration of 30 g./L. The main purpose of the potassium chloride was to facilitate anode corrosion, and the amount added was increased from the minimum only if anode dissolution was not progressing satisfactorily.

The corrosive nature of the hot solutions made filtration a problem, and also resulted in attack on the immersion heaters. Nickel deposition proceeded relatively satisfactorily, but nodules or spots occurred

during copper plating, particularly when the deposit was brushed with a glass brush (brushing the copper coating with glass brushes or agate blocks during deposition refined the grain structure). Copper was electrodeposited onto nickel with much less difficulty than nickel onto copper.

Fabricating Qualities of Nickel-Plated Steel Strip

F. P. ROMANOFF: 'Fabricating Qualities of Pre-Finished Metals.'

Metal Finishing, 1960, vol. 58, Feb., pp. 55–62.

The fabrication of articles from 'pre-finished' metals (i.e., from plated sheet or strip) has revealed the need for some means of quantitatively determining the fabricating characteristics of the basis-metal/electrodeposit composite. In the present paper details are given of the procedures and testing equipment used in, and the data derived from, tests to determine the influence of various electrodeposited metals on the fabrication properties of sheet or strip steel.

Since the speed of deformation is often influential in this respect, a test simulating a punch-press operation was desired. In the investigations described use was therefore made of the dynamic ductility machine described in the A.S.T.M. Standard for Rolled Zinc (*Designation B*-69-39). This machine, which was found adaptable to routine testing, consists essentially of a modified inclinable punch press fitted with a micrometer-adjustable $\frac{3}{8}$-in.-diameter ball die, and a toggle-operated clamping device. The test specimen is 3·5 in. (8·75 cm.) wide and 18 in. (45 cm.) or more long. A series of cups is made along the middle of the strip: if the first cup does not fracture, the plunger is advanced, in increments of 0·005 in. (0·125 mm.), until rupture occurs. It was found that some steels exhibit considerable flow or 'necking' which persists over an increase in depth of 20 to 30 mils beyond the first necking. Some steels exhibited no 'necking', and a 5-mil increase in cup depth produced rupture. The end point found most satisfactory on the pre-finished and basis steel was the cup depth just before the occurrence of the first detectable fracture of the base.

The procedure employed for routine checking of pre-finished sheet is outlined, and, as a means of indicating the effects of plating and basis-metal variables, typical results are presented for nickel- and brass-plated steel.

In subsequent sections the author discusses data illustrating the influence of buffing on the fabrication of steel sheet plated with various types and thicknesses of nickel or copper. The effects of 'ageing' were assessed by testing either on the same day as, or several days after, plating. Some tests were conducted to study the ductility of steel sheet as a function of the electrodeposition of various thicknesses of chromium directly onto one or both surfaces of the steel: in others the ductility of nickel-plated steel was determined as a function of chromium plating.

Consideration of the data presented leads the author to conclusions which he summarizes as follows:

'1. The dynamic ductility machine serves to indicate the effect of electrodeposits of copper, nickel, chromium and brass on the fabricating properties of sheet or strip steel.

'2. Ductile brass, copper and nickel electrodeposits tend to increase the depth of draw of low-carbon, cold-rolled strip or sheet steel.

'3. Unbuffed copper, brass and nickel deposits tend to reduce the depth of draw of the basis steel immediately after plating.

'4. Aging tends to increase the depth of draw of unbuffed ductile deposits to values approaching that of buffed deposits.

'5. Buffing usually improved the depth of draw of deposits on low-carbon steel, except in the case of some deposits from bright or badly contaminated baths.

'6. The thickness of ductile deposits does not affect the ductility of the composite.

'7. Non-ductile deposits of copper, brass, bronze, or nickel tend to reduce the ductility of the composite below that of the unplated steel.

'8. The depth of cup of non-ductile deposits tends to vary inversely with the thickness.

'9. Chromium can be applied to ductile nickel deposits without embrittlement. Thickness of chromium deposits up to 50 millionths inch has no effect on ductile nickel on low-carbon steel.'

NON-FERROUS ALLOYS

Production and Properties of Lead-Cemented Alloys

See abstract on p. 154.

Brazing of Nickel-containing Alloys

See abstract on p. 157.

Check Analysis Limits for Nickel-base Alloys

See abstract on p. 151.

NICKEL-IRON ALLOYS

Activity of Carbon in Iron-Nickel Alloys at 1000°C.

R. P. SMITH: 'Activity of Carbon in Iron-Nickel Alloys at 1000°C.'

Trans. Metallurgical Soc., Amer. Inst. Mining, Metallurgical and Petroleum Engineers, 1960, vol. 218, Feb., pp. 62–4.

The carbon content of iron-silicon and iron-manganese alloys at fixed activities of carbon has

been previously reported by the author (*Jnl. Amer. Chemical Soc.*, 1948, vol. 70, p. 2724). The investigation now described has extended the scope of this work to the iron–nickel system, all the alloys of which are austenitic at 1000°C. and therefore permit study of a wider range of alloy composition within a single phase region.

The carbon content of high-purity iron, high-purity nickel, and of eight iron–nickel alloys ranging in nickel content from 3·98 to 99·4 per cent., was determined, at four fixed activities of carbon, by equilibration with each of four CO/CO_2 atmospheres at 1000°C. At a fixed activity of carbon, the carbon content decreased with increase in the nickel content to about 72 per cent. nickel, and then increased with rise in the nickel content to 100 per cent. This minimum solubility, corresponding to a maximum activity coefficient, is not considered explicable in terms of simple 'pseudo-chemical' theory.

Influence of Temperature on the Properties of 'Alcomax III'

A. G. CLEGG and M. McCAIG: 'The Effect of Temperatures of 650°C. and 700°C. on the Magnetization and Properties of 'Alcomax' Magnets.'

*Brit. Electrical and Allied Industries Research Assocn., Tech. Report N/T*86, 1960; 4 pp.

The experiments reported were carried out (in view of interest in the use of permanent magnets at elevated temperatures, and of the desirability, in some applications, of assembling magnets by die casting or brazing) to determine the effects, on the magnetization of 'Alcomax III'*, of exposure at 650° and 700°C. for times ranging from 5 minutes to 21 hours.

The results of this and previous investigations indicate that 'Alcomax' magnets should not be exposed to temperatures higher than 550°C. if stability of magnetization is required.

Exposure for not more than 5 minutes at 650°C. during manufacture prior to magnetization should not result in more than a 2 per cent. deterioration in performance, and the magnets can, it is considered, be used for short periods of an hour or so at 700°C., and for more than 21 hours at 650°C., provided that a performance of about 50 per cent. worse than that obtainable at room temperature can be tolerated.

Brazing of Nickel-containing Alloys

See abstract on p. 157.

CAST IRON

Properties and Applications of 'S.G. Ni-Resist' Austenitic Cast Irons

A. B. EVEREST and O. NICKEL: 'Spheroidal-Graphite Types of 'Ni-Resist' Austenitic Cast Iron.' Translation of a paper published in *Konstruktion*, 1959, vol. 11, No. 10, pp. 390–5.

Foundry Trade Jnl., 1960, vol. 108, Apr. 28, pp. 515–22.

The development, after the Second World War, of treatments capable of producing grey cast irons with spheroidal (as opposed to flake) graphite enabled the production (mainly by magnesium treatment) of nickel-chromium spheroidal-graphite austenitic irons which combine all the advantages of the original flake-graphite grades with the improved mechanical properties associated with the spheroidal-graphite structure. This series of irons, known under the generic of 'S.G. Ni-Resist', offers such properties as corrosion-resistance, heat- and oxidation-resistance, high- or low-thermal-expansion characteristics, resistance to wear and galling, non-magnetic properties, improved impact-resistance, and a tensile strength about twice that of the corresponding flake-graphite grades. In this paper the properties of the various grades of 'S.G. Ni-Resist' are summarized, and some indication is given of the wide range of applications for which such irons have proved suitable.

The composition ranges and the physical and mechanical properties of the various grades of 'S.G. Ni-Resist' are indicated in tables which are reproduced on p. 142.

Types D2 and D2B are recommended for applications requiring resistance to corrosion, erosion, wear, and temperatures up to 760°C. Both grades are characterized by a high thermal expansion.

Type D2C is used where high ductility is important and where medium heat- and corrosion-resistance is required (e.g., for components which have to be welded). This grade retains its properties well at low temperatures, and is recommended for use at temperatures below 0°C.

The good frictional properties of Type D3A renders it suitable for applications involving exposure to wear and corrosion and requiring medium thermal-expansion characteristics. The higher nickel, chromium and silicon contents of Type D4 are associated with particularly high resistance to corrosion, erosion and oxidation, while Types D5 and D5B, characterized by low thermal expansion, are suitable for use in precision machine components, scientific instruments, glass moulds, etc. Type D5 retains its properties at temperatures as low as −196°C.

Welding and machining characteristics are considered, and attention is drawn to heat-treatments employed for stress relief, to improve machinability when hardness values are excessive, to obviate the risk of distortion during high-temperature service, and to ensure maximum stability in machined castings. The authors then discuss the degree to which the irons satisfy such service requirements as resistance to wear, galling, erosion, cavitation, corrosion and oxidation, and the first part of the paper concludes with sections concerned with mechanical properties at low and elevated temperatures, and with thermal-expansion and magnetic characteristics.

The following applications are stated to be typical of those found by the 'S.G. Ni-Resist' series of cast iron: (*a*) those requiring good corrosion-resistance, (*b*) those requiring wear-resistance in combination with other special properties, (*c*) those requiring

*Composition: cobalt 25, nickel 13·5, aluminium 8, copper 3, niobium 0·8, per cent., remainder iron.

Composition Ranges of 'S.G. Ni-Resist' Irons

(*See abstract on p. 141*)

Type	C %	Si %	Mn %	Ni %	Cr %
D2	3·00 max.	1·75-3·00	0·70-1·00	18·00-22·00	1·75-2·50
D2B	3·00 max.	1·75-3·00	0·70-1·00	18·00-22·00	2·75-4·0
D2C	2·90 max.	2·00-3·00	1·80-2·40	21·00-24·00	0·50 max.
D3	2·60 max.	1·50-2·80	0·50 max.	28·00-32·00	2·50-3·50
D3A	2·60 max.	1·50-2·00	0·50 max.	28·00-32·00	1·00-1·50
D4	2·60 max.	5·00-6·00	0·50 max.	29·00-32·00	4·50-5·50
D5	2·40 max.	1·50-2·75	0·50 max.	34·00-36·00	0·10 max.
D5B	2·40 max.	1·30-2·75	0·50 max.	34·00-36·00	2·00-3·00

Mechanical and Physical Properties of 'S.G. Ni-Resist' Irons

(*See abstract on p. 141*)

Type	D2	D2B	D2C	D3	D3A	D4	D5	D5B
Tensile strength tons/sq. in.: kg./mm.²:	24·5-31 38·5-49	26-31 41-49	24·5-29·0 38·5-45·5	24·5-30·0 38·5-47·0	24·5-29·0 38·5-45·5	27-32 42·5-50·5	24·5-27·0 28·5-42·5	24·5-29·0 28·5-45·5
0·2% proof stress tons/sq.in.: kg./mm.²:	14-16 22-25·2	14·7-16·5 23·1-26	13·0-15·7 20·5-24·7	14·7-16·5 23·1-26	14·5-17·5 22·8-27·5	17·0-19·5 26·7-30·7	13·2-15·7 20·8-24·7	15·7-18·7 24·7-29·5
Elongation on 2 in.	8-20	7-15	20-40	7-18	13-18	1·5-4·0	20-40	5-10
Elastic modulus (lb./sq. in. × 10^{-6})	16·5-18·5	16·5-19·0	15	13·5-14·5	16·0-18·5	13	16-20	16·0-17·5
Brinell hardness	140-200	150-210	130-170	140-200	130-190	170-240	130-180	140-190
Specific gravity	7·41	7·45	7·41	7·45	7·45	7·45	7·68	7·72
Melting point (°C.)	1,240	1,260	1,240	1,240	1,240	1,205	1,240	1,240
Coefficient of thermal expansion per °C. (20°-200°C.) × 10^{-6}	18·7	18·7	18·4	12·6	12·6	14·4	5·0	5·0
Magnetic properties	Non-magnetic	Slightly magnetic	Non-magnetic	Magnetic	Magnetic	Slightly magnetic	Magnetic	Magnetic

heat-resistance and strength at high temperature, (*d*) those requiring non-magnetic properties, and (*e*) applications involving special thermal-expansion characteristics. In the second part of the paper the authors discuss each of these applications in relation to the range of components for which the irons have been found suitable. Photographs of typical castings supplement the authors' comments.

Nickel-containing Filler Rod for the Gas-Welding of S.G. Iron

R. H. T. DIXON and D. R. THORNEYCROFT: 'Filler Rod for the Gas-Welding of S.G. Iron.'

Foundry Trade Jnl., 1960, vol. 108, May 12, pp. 583-9.

Spheroidal-graphite cast iron has been successfully welded by various techniques, including the metal-arc, argon-shielded and submerged-arc welding

processes. In certain cases, however, the oxy-acetylene process would, were an adequate welding rod available, provide the most suitable welding method: e.g., for deposition of a small quantity of S.G. iron in the reclamation of castings in the foundry, for replacement of S.G. iron surfaces worn in service, or for the fabrication of composite assemblies in which it is important that the composition, structure, and properties of the weld metal be closely similar to those of the parent material. Although previous investigations of the gas-welding process had shown that joints of reasonable strength could be obtained by using filler-rods of a modified S.G.-iron composition, it had been found that the attainment of these properties entailed special precautions during welding, and the need to improve both the deposition characteristics of the rod and the properties of the deposited weld metal was generally acknowledged. This paper traces the development of a nickel-bearing S.G.-iron filler rod which substantially meets these requirements.

On the assumption that a rod suitable for welding S.G. iron should contain graphitizing elements (in order to prevent formation of eutectic carbide during welding), a number of filler-rods were made with compositions in the following range: nickel 1–7, aluminium 0·05–1, carbon 3–4, silicon 2–3, calcium 0–0·01, cerium 0–0·05, magnesium 0·04–0·1, per cent. The usual carbide-forming elements were excluded; in particular, chromium and manganese were kept below 0·1 per cent., although the effect of manganese was subsequently investigated over the range 0·1 to 0·3 per cent. In initial tests with these rods single beads were deposited on the surface of S.G. iron, and butt-welds were made in ½-in. (1·25 cm.) plate. Examination of the microstructures of the deposits established the importance of controlling the content of the elements studied. On the basis of the data obtained, the following preferred composition was selected for use in subsequent tests: carbon 3·8, silicon 2·5, nickel 5, manganese 0·1, aluminium 0·06, magnesium 0·06, per cent. Silicon was restricted to 2·5 per cent. because of its known adverse effect upon impact properties. Aluminium proved to be an effective deterrent to carbide formation, but in amounts exceeding 0·2 per cent. it seriously impaired the flow of the weld metal, even when special fluoride-bearing fluxes were used. The results indicated, however, that the presence of a small percentage of aluminium strengthened the effect of nickel in preventing formation of carbide.

Using a rod of the preferred composition, butt joints were made in ½-, 1- and 2-in.-thick (1·25-, 2·5- and 5-cm.-thick) plate of pearlitic S.G. iron. Standard welding procedures were employed, including the use of a proprietary gas-welding flux normally used for cast iron. Tensile tests were carried out on the welds and on weld-metal specimens, and the impact properties of welds made with rods of both normal S.G. iron and the modified composition were also determined. Weldments were subjected to metallographic examination, and response to flame-tempering treatment was studied.

The data presented show that the new filler-rod has attractive deposition characteristics and produces weld joints with consistently high mechanical properties. The tensile strength of as-welded joints made in pearlitic irons was 42½–46½ t.s.i. (70–73 kg./mm.²) on ½-in. plate, 37–40 t.s.i. (58–63 kg./mm.²) on 1-in. plate, and 38–41½ t.s.i. (60–65 kg./mm.²) on 2-in. plate. Impact-resistance, and response to the full ferritizing heat-treatment, were satisfactory. The new rod will thus, it is considered, meet requirements where it is essential that the composition and mechanical and chemical properties of welds be similar to those of the parent S.G. iron. Steps are being taken to initiate industrial trials of the new welding rods, both in relation to fabrications incorporating castings and to the reclamation of defective castings.

CONSTRUCTIONAL STEELS

Creep Testing of Steels: British Standard

See abstract on p. 146.

Determination of Rupture Strength of Steel: British Standard

See abstract on p. 146.

Age-Hardenable Nitriding Steel

W. S. MOUNCE and A. J. MILLER: 'A Nitriding Steel That Age Hardens.'

Metal Progress, 1960, vol. 77, Feb., pp. 91–4.

This article gives details of the development and properties of a steel which can be age-hardened, by heat-treatment in the range 950°–1050°F. (510°–570°C.), to a tensile strength of 200,000 p.s.i. minimum (89·5 t.s.i.; 140·5 kg./mm.²), and which can be nitrided, in the same temperature range, to produce a case equivalent to that formed on standard 'Nitralloy' steels.

Compositional limits are: carbon 0·02–0·25, manganese 0·25–0·45, nickel 4·75–5·25, aluminium 1·8–2·2, chromium 0·4–0·6, molybdenum 0·2–0·3, vanadium 0·08–0·15, per cent. Full strength and toughness are achieved by normalizing at 1700°F. (925°C.), oil quenching from 1650°F. (900°C.), solution-treating at 1275°F. (690°C.), and ageing at 1050°F. (565°C.) for not less than 5 hours. Graphs illustrate the influence, on the hardness, impact-resistance and tensile properties of the steel, of ageing for various times in the range 950°–1100°F. (510°–595°C.). The nitriding characteristics of the steel are indicated, in terms of response to four nitriding cycles, in the table reproduced on p. 144.

The resistance to tempering exhibited by both core and nitrided case is stated to render the steel particularly suitable for use in cams, gears, shafts and bearings required to exhibit fatigue- and wear-resistance at temperatures up to 1000°F. (540°C.).

Treatment	Case Depth	Rockwell Hardness	
	in.	Case	Core
A. Floe process, 875°F., 6 hr.; 1025°F., 30 hr.	0·015	15-N-93	C-45
B. 'Chapman Ni-20'* 1020°F., 20 hr.	0·012	15 N-93	C-46
C. 'NitroCycle' pressure nitriding† 7·5 g./sq. ft. NH_4, 1000°F., 15 hr.	0·010	15 N-93·3‡	C-45·5‡
53 g./sq. ft. NH_4, 1000°F., 21 hr.	0·015	15 N-93·3‡	C-45·5‡
D. Gas nitrided, 975°-1000°F. 24 hr.	0·012	15 N-92·5	C-49·5
48 hr.	0·015	15 N-93·2	C-49·2
48 hr. (0·0002-in. white layer)	0·020	15 N-91·4	C-50

* Licensed by Chapman Valve Company.
† Patent process, Oil Well Supply Div., U.S. Steel Corporation.
‡ By conversion from diamond pyramid hardness.

Influence of Alloying Additions on the Tempering Characteristics of Low-Carbon Bainitic Steels

K. J. IRVINE and F. B. PICKERING: 'The Tempering Characteristics of Low-Carbon Low-Alloy Steels.'
Jnl. Iron and Steel Inst., 1960, vol. 194, Feb., pp. 137–53.

Interest in the tempering characterists of low-carbon low-alloy steels has been reflected in investigations of both bainitic and martensitic grades. The beneficial effects of tempering on the mechanical properties of bainitic steels, particularly at the higher strength levels, induced the authors to make a systematic study of the tempering characteristics of these steels. The inclusion within the scope of the investigation of both bainitic and martensitic specimens enabled determination of the effects, on tempering characteristics, of the initial structure of the steel. The work described is intended to form the background to future investigation of alloy steels, and subsequent papers will indicate how a knowledge of the effect of alloying elements on the tempering characteristics of low-carbon steel can be used in the development of alloy steels.

The basis composition (0·15 carbon–0·5 molybdenum–boron), selected with the aim of obtaining bainitic structures over a wide range of alloying additions, was modified, in a series of steels, to permit investigation of the effects of the following alloying additions: carbon, manganese, nickel, chromium, molybdenum, tungsten, vanadium and titanium. The influence of the initial hardness of the steel was also taken into account. Full details are given of the steels studied and the test procedures employed, and the paper includes numerous photomicrographs illustrating structures after various tempering treatments.

It was possible to distinguish clearly between the effects of molybdenum, tungsten, vanadium and titanium (which produced marked secondary hardening) and those of manganese and nickel (which did not).

The main feature of the microstructural examination was that the elements which resulted in secondary hardening produced separately nucleated carbides, whereas manganese and nickel did not form a separate carbide. Chromium was intermediate in its influence: although it produced some separately nucleated carbide the secondary- hardening effect was negligible. In any steel which exhibited a marked secondary-hardening reaction the amount of secondary hardening decreased, and the rate of secondary hardening increased, as the initial hardness of the structure increased.

Development of High-Strength Steels by Working of Metastable Austenite

F. R. MORRAL: 'Development of High-Strength Steels by Working of Metastable Austenite.'
Defense Metals Information Center, Memorandum 39, Nov. 30, 1959; 20 pp.

Work being carried out to improve the properties of high-strength low-alloy steels may be classified within three main categories:

(1) Modification of the composition of standard steels to improve hardenability and tempering characteristics.

(2) Development of tempering treatments capable of improving strength properties.

(3) Incorporation of deformation into the heat-treatment procedure.

This memorandum comprises an attempt to collate the findings of the most interesting and promising current work falling within category (3): i.e., the development of tensile strengths in the region of 340,000 p.s.i. (152 t.s.i.; 239 kg./mm.^{2}) by deformation, in the metastable-austenite condition, prior to quenching and tempering.

The review, based on published work and findings communicated personally to the author, includes numerous data illustrating the effects of such hot-cold-working treatment on the properties of the few steels investigated up to the date of the memorandum: i.e., steels of nickel, nickel-chromium, nickel-chromium - molybdenum, chromium, chromium - molybdenum, chromium - molybdenum - vanadium, and chromium-molybdenum-nickel-vanadium type.

It is concluded that in hot-cold-worked high-strength steels, as in those conventionally heat-treated, strength tends to increase, and ductility to decrease, with increase in the carbon content. The greater the deformation of the metastable austenite, the better the yield and tensile strength: tensile strengths of 400,000 p.s.i. (178 t.s.i.; 281 kg./mm.^{2}) have been obtained with deformations (during rolling) of the order of 90 per cent. reduction in cross-section. Steels containing 0·3–0·4 per cent. carbon, to which conventional heat-treatment imparts good properties,

can be improved by deformation of metastable austenite and are deemed the most suitable for the aircraft and missile industries. The effects of individual alloying elements on the behaviour of the deformed metastable austenite is not known, and the tempering sequence will depend on the specific steel.

Development of suitable plant techniques is considered a prerequisite to commercial exploitation of hot-cold-working procedures. Suggestions for future development and research work are listed.

Fatigue of Welded Joints in High-Strength Steels

J. E. STALLMEYER and W. H. MUNSE: 'Fatigue of Welded Joints in High-Strength Steels.'

Brit. Welding Jnl., 1960, vol. 7, Apr., pp. 281–7.

The main advantages offered by the high-strength low-alloy steels are manifested in the increase in the design stress which the higher yield point of such steels permits, and (due to the thinner sections required) in the corresponding savings in weight, and hence in material and often fabrication costs. In the case of components subjected in service to stress cycling, however, the maximum stress permitted by design specifications is usually lower, and this reduction can offset any previous economic advantage. Awareness of the importance of the fatigue-resistance of such steels has therefore increased. The present paper, written in the light of this awareness, reviews data available on the fatigue strength of various types of welded joint in alloy structural steel.

Steels regarded of relevance, and to which therefore the discussion relates, exhibit ultimate strengths in the range 75,000–150,000 lb./sq. in. (33·5–67 t.s.i.; 53–106 kg./mm.^{2}). The data reviewed were derived from fatigue tests on three types of joint: transverse butt-welded, longitudinal butt-welded, and fillet-welded joints. The fatigue behaviour of the different joints are considered in terms of the properties of the basis metal, joint preparation, stress variations, and the location of failures.

On the basis of their evaluation of the available data, the authors conclude that: 'In general, the fatigue strength, on a zero-to-tension stress cycle, of transverse butt-welded joints in low-alloy structural steels does not differ significantly from the strength of the same type of joint in ordinary mild-grade structural steel when the comparison is made at 2×10^6 cycles. This is particularly true if the joint in the ordinary structural steel is made with a low-hydrogen electrode. The particular advantage of the low-alloy steels would appear to occur when the stress cycle covers a small range from some value of tension stress to a maximum tensile stress. In this case it is possible to use a greater value of the maximum stress for the low-alloy steels than for the ordinary structural steels.

'The improvement of fatigue life of transverse butt-welded joints obtained by treatment of the weld, such as removal of the weld reinforcement or grinding of the plate surface at the joints, is dependent on the internal structure of the weld. If there are significant weld defects within the weld metal, there may be no improvement as a result of such treatment.

'For the longitudinal butt-welded joints there is an improvement in fatigue strength over ordinary structural steel if a low-alloy steel is used. This is to be expected, since the fatigue properties of this type of joint are largely a function of the base-metal properties. In almost all cases, the fatigue strength of this type of connection approaches very nearly that of the parent-plate. As for the transverse butt-welded joints, any improvement expected from the removal of the weld reinforcement is dependent on the internal structure of the weld.'

Cold-Cracking in Low-Alloy-Steel Welded Joints

J. T. BERRY and R. C. ALLAN: 'A Study of Cracking in Low-Alloy-Steel Welded Joints.'

Welding Jnl., 1960, vol. 39, Mar., pp. 105S–16S.

This paper outlines the preliminary findings of a comprehensive investigation of the rôles played by hydrogen and other metallurgical factors in the cracking of welded joints.

The authors' report of their experimental work is preceded by a detailed review of the literature (reference is made to a *bibliography* of 92 items), and an attempt is made to interpret, on the basis of the information available, the modes of cracking in weldments.

In the experimental phase of the study, use was made of a controlled-atmosphere welding chamber in which controlled-arc welding could be performed in various partial pressures of hydrogen and argon, enabling maintenance of hydrogen contents of up to 16·5 p.p.m. of weld metal. The welding wire employed provided a weld metal of the following composition: carbon 0·15, manganese 1·0, nickel 1·0, molybdenum 0·4, chromium 0·1, per cent. The two steels principally used as basis metals contained, respectively, carbon 0·2, manganese 0·5, nickel 4·0, chromium 2·0, molybdenum 0·5, per cent., and carbon 0·15, manganese 1·0, nickel 1·0, chromium 1·0, molybdenum 0·5, per cent. Two types of weld test were used: bead-on-plate and controlled-thermal-severity tests.

The results of this experimental phase are summarized in the form of a series of cracking diagrams, and the findings of a metallographic study of welds which exhibited cracking are also discussed.

The metallographic data obtained in the present study conform with the structural requirements of the theory of cold cracking advanced as a result of the authors' literature survey, i.e., the presence of a 'plastic' and a 'non-plastic' phase, each of relatively high ultimate strength, was observed, together with a 'lath' or grain-boundary region of somewhat lower strength. The first phase is typified by higher-temperature bainitic ferrite, the second by higher-carbon martensite. In conjunction with local stress amplification, these structural features participate in propagation of cracking. (The term 'lath' or 'grain-boundary region' embraces such discontinuities

as inclusions, films and small pre-existing hot cracks.) In the main the principal structural features will depend upon the variable of cooling rate.

Hydrogen is regarded as assuming its major rôle during crack propagation (i.e., as soon as an embryo crack is formed), though rôles which affect the rate of work hardening and transformation should not, it is considered, be overlooked. The effective hydrogen content is, however, itself dependent upon the cooling rate, both from the point of view of hydrogen loss to the atmosphere and basis metal, and from that of the time available for the hydrogen to consolidate embryo cracks. Although both variables will promote crack propagation, it would appear that the cooling rate, because of its major effects on transformation, is the more instrumental.

Use of Nickel-Copper Alloy Steel for Bridges

W. D. MOGERMAN: 'Corrosion-Resistance—Another Reason to Specify Low-Alloy Steel for Bridges.'

Consulting Engineer, 1959, Oct.

Reprint issued by THE INTERNATIONAL NICKEL COMPANY, INC.; 8 pp.

High-strength low-alloy steels have long been accepted as materials of construction for bridges: indeed, in terms of economics alone, many modern designs could not have been put into effect without their use, since many structural codes call for strengths that would, in carbon steel, require the use of unreasonably heavy sections. It has, however, been calculated that the expense involved in maintenance and corrosion prevention ultimately amounts to a figure nearly twice the original construction costs of the bridge, and it is not with the mechanical properties, but with the corrosion-resistance of low-alloy steels that the present paper is concerned.

A survey of the metallic materials of construction used for bridges since the building of the first cast-iron bridge leads to a discussion of the relevant properties obtainable in low-alloy steels, and, in particular, of the advantages offered by those high-strength steels which not only satisfy the requirements of *A.S.T.M. Specification A-242*, but also possess the enhanced corrosion-resistance conferred by nickel and copper alloying additions.

Work on these steels (to which the author makes due reference) has shown that they are more corrosion-resistant than carbon steels, that coatings of paint are more adherent and last 20–80 per cent. longer than on carbon steel, and that they are extremely resistant to 'corrosion creep' (accelerated failure of the paint film as a result of local damage).

The types of application which nickel-copper high-strength steels have found in bridge construction are briefly outlined, and mention is made of the economic advantages which might accrue from their enhanced corrosion-resistance and the longer intervals between painting schedules associated with their use (in 1946 it cost approximately $300,000 to repaint the George Washington Bridge).

HEAT- AND CORROSION-RESISTING MATERIALS

Creep Testing of Steels: British Standard

BRITISH STANDARDS INSTN.: 'Mechanical Testing of Steel at Elevated Temperatures. Part 3. Non-interrupted Creep Testing.'

B.S. 3082: *Part* 3: 1959; 9 pp. Price 4/-.

This British Standard, authorized by the Iron and Steel Industry Standards Committee, is one of a series dealing with the mechanical testing of steel at elevated temperatures: for abstract of Part 2, see *Nickel Bulletin*, 1959, vol. 32, No. 11, p. 330. The Standard covers 'those tests in which the strain is measured under tensile creep stresses and in which the period of testing does not exceed 10,000 hours.'

Creep-testing terminology and symbols are defined, and requirements are specified with respect to: test pieces; determination of creep strain; heating apparatus; measurement of temperature; heating of test piece and soaking period; testing machine; loading; temperature and creep readings; and presentation of results.

Determination of Rupture Strength of Steel: British Standard

BRITISH STANDARDS INSTN.: 'Procedures for Obtaining Properties of Steel at Elevated Temperatures. Part 2. Rupture Strength.'

B.S. 3228: *Part* 2: 1960; 7 pp. Price 3/-.

This British Standard, authorized by the Iron and Steel Industry Standards Committee and covering determination of the properties of steel at elevated temperatures, specifies the procedure for 'obtaining the range of rupture strength at elevated temperatures for a selected steel in lengths of time up to 100,000 hours'.

Requirements are laid down with respect to: test method; selection of samples; number of samples and test conditions; presentation of results.

Creep Strength of Nickel-containing Heat-Resisting Materials in the Range 1050°-1200°C.

H. C. CHILD, A. B. COLLIER and C. F. WEST: The Creep Strength of Some High-Temperature Alloys at 1050°-1200°C.'

Metallurgia, 1960, vol. 61, Feb., pp. 51–4.

Although the stress-rupture properties of austenitic steels and nickel- and cobalt-base alloys have been exhaustively studied as a function of temperatures up to 1000°C., little information is available on creep properties at higher temperatures: the result partly of the few commercial applications involving such very high temperatures, and partly of the extensometry difficulties associated with creep testing in the relevant temperature range. In view of the rise in the operating temperatures of gas-turbine engines, and the considerable use being made of austenitic steels for furnace parts and heat-treatment jigs, the authors deemed it of value to determine the creep strength of typical

grades of wrought and cast austenitic material (*see* table below) in the temperature range 1050°–1200°C.

Creep testing was carried out at strains of up to 1 per cent., and for times of up to 1000 hours. Use of a cantilever-bend specimen obviated the difficulties inherent in tensile testing. Where the material under study exhibited adequate oxidation-resistance the tests were conducted in air; otherwise they were carried out in an atmosphere of static argon.

Data tabulated in the paper indicate the stresses required to produce 0·1, 0·2, 0·5 and 1 per cent. plastic strains in 30, 100, 300 and 1000 hours. The creep characteristics of the various materials are compared on the basis of the stress to produce 1 per cent. plastic strain in 100 hours. The results demonstrate that all the alloys exhibit a similar decrease in strength with rise in temperature. Of interest is the finding that, in general, the cast alloys were very much more creep-resistant than their wrought equivalents. It has been known for some time that the creep and rupture properties of cast materials tend to become superior to those of wrought as the temperature is raised, but at 1100°C. and higher this trend becomes very pronounced: the creep strength of the as-cast 'R.22' alloy is, for example, more than 700 per cent. higher than that of the material in the wrought condition.

'G.39', a nickel-base alloy developed for precision-cast turbine rotor blades, proved the strongest of the alloys tested: its creep strength, based on the criterion of 1 per cent. strain in 100 hours, was approximately 1 t.s.i. at 1100°C. (i.e., 1400 p.s.i. greater than that of any other of the materials studied). This alloy has been successfully used as a high-temperature mandrel material for the hot bending of stainless-steel tubes.

Resistance of 'Udimet 500' and 'DCM' to Thermal Fatigue

F. J. MEHRINGER and R. P. FELGAR: 'Low-Cycle Fatigue of Two Nickel-base Alloys by Thermal-Stress Cycling.'

Amer. Soc. Mechanical Engineers, 1959, *Paper No.* 59-*A*-58; 10 pp.

In recent years work on the designing of components for applications involving fluctuations in temperature has focused attention on the problem of thermal fatigue, and, in any evaluation of the properties of a new material, resistance to thermal fatigue now receives as much study as static high-temperature properties. The aim of the present paper is two-fold: to report the results of thermal-strain-cycling tests on 'DCM' and 'Udimet 500' heat-resisting casting alloys; and to discuss problems associated with the testing of materials of such relatively low ductility.

The percentage composition of the 'DCM' alloy and the two heats of 'Udimet 500' used in the study are tabulated on p. 148.

After heat-treatment, the hardness of the 'Udimet 500' specimens was in the range Rc35–37; the 'DCM'

Composition of Heat-Resisting Materials

(*See abstract on p. 146*)

	C %	Mn %	Si %	Ni %	Cr %	W %	V %	Mo %	Co %	Fe %	Other Elements %
'R.29'	0·12	0·6	0·9	0·4	21·0	—	—	0·45	—	Bal.	Ti 0·35
'R.12'	0·2 max.	0·55	1·25	1·25	27·0	0·4	—	0·7	—	Bal.	Ti 0·4
'R.22'	0·21	0·9	1·0	14·0	21·0	2·8	—	—	—	—	—
'R.40'	0·21	0·8	1·0	11·5	22·5	1·0	—	—	—	Bal.	—
'R.23'	0·12	1·1	1·75	22·5	25·5	—	—	—	—	Bal.	—
'R.13'	0·8 max.	0·5	1·75	61·0	16·5	—	—	—	—	—	—
'G.39'	0·5	1·0	1·0	60·0	20·0	3·0	—	3·0	—	Bal.	Nb 1·5 Ta 1·5
'Nimonic 75' ..	0·12	1·0 max.	1·0 max.	Bal.	19·5	—	—	—	—	5·0 max.	Ti 0·4 Cu 0·5 max.
'Nimonic 80' ..	0·1 max.	1·0 max.	1·0 max.	Bal.	19·5	—	—	—	2·0 max.	5·0 max.	Ti 2·2 Al 1·2
'Nimonic 90' ..	0·1 max.	1·0 max.	1·5 max.	Bal.	19·5	—	—	—	18·0	5·0	Ti 2·2 Al 1·4
'G.19'	0·4	0·6	1·0	13·0	19·0	2·5	—	2·0	10·0	—	Nb 3·0
'G.42B'	0·25	1·0	0·40	15·0	19·5	2·2	2·2	2·3	25·0	—	Ti 0·85 Nb 2·3
'G.32'	0·3	0·8	0·5	12·5	19·0	—	2·8	2·0	45·0	—	Nb 1·3

specimens exhibited a hardness of Rc40. The latter alloy was thermally cycled from minimum temperatures in the range 150°–500°F. (65°–260°C.) to maximum temperatures in the range 1000°–1600°F. (540°–900°C.); the former was cycled using minimum temperatures of 200°–400°F. (95°–205°C.) and maximum temperatures of 900°–1700°F. (480°–925°C.). Full details are given of the test apparatus, procedure and specimens employed.

The plastic strain causing failure was so small that in neither case could it be determined with sufficient accuracy for use as a parameter in evaluating the resistance of the alloys to thermal fatigue. It is suggested, however, that a plot of stress range versus the logarithm of the number of cycles to failure could be used to describe thermal-fatigue behaviour.

Element	'DCM'	'Udimet 500'	
		Heat 3V-13	Heat 3V-14
Cr	15·25	18·95	19·20
Co	—	19·30	18·60
Fe	4·50	0·39	0·52
Mo	5·20	4·10	4·15
Al	4·55	3·03	3·10
B	0·082	0·008	0·006
Zr	—	0·11	0·03
Ti	3·60	2·90	3·08
Cu	0·10	0·06	0·10
C	0·06	0·08	0·06
Si	0·05	0·05	0·03
Mn	0·10	Trace	0·10
S	0·005	0·008	0·008
Ni	Bal.	Bal.	Bal.

Relationship between Structure and Mechanical Properties in 'Udimet 500'

M. KAUFMAN and A. E. PALTY: 'The Relationship of Structure to Mechanical Properties in 'Udimet 500'.'

Trans. Metallurgical Soc., Amer. Inst. Mining, Metallurgical and Petroleum Engineers, 1960, vol. 218, Feb., pp. 107–16.

The mechanical properties of heat-resisting precipitation-hardenable alloys are, under given test conditions, governed principally by grain-size and by the phases present, the distribution of these phases, and the degree to which they are influenced by temperature and time at temperature. The work reported was carried out with the aim of increasing the relatively little information available on the relationship between mechanical properties and structure, and, in particular, to correlate structure and grain-size with the hardness, stress-rupture life, bend ductility and fatigue strength exhibited by the high-temperature alloy 'Udimet 500'.

The composition of the two heats of alloy studied fell within the normal specification limits*. The phases present as a result of the various ageing treatments to which the specimens were subjected were determined by microscopical examination and X-ray-diffraction measurements. This work led to the determination of the following four phases:

Ti(C,N) which was present under all conditions up to 2250°F. (1230°C.), but appeared to decrease in favour of $M_{23}C_6$ in the range 1550°–1800°F. (840°–980°C.).

$M_{23}C_6$, a grain-boundary precipitate which is taken completely into solution in the range 1900°–1975°F. (1035°–1075°C.), and precipitates heavily at 1550°–1800°F. (840°–980°C.). It agglomerates at 1900°F. (1035°C.).

$Ni_3(Al, Ti)$, present at 1400°F. (760°C.) in the form of a fine uniformly-distributed general precipitate which gradually coarsens as the temperature approaches the solution temperature (1975°–2000°F.: 1075°–1095°C.). Its titanium content is higher at higher temperatures.

'*X*' which, possibly a titanium-rich phase, appears under the same conditions as Ni_3(Al, Ti) and is indistinguishable in its behaviour.

The second stage of the investigation was undertaken in an attempt to establish some relationship between the mechanical properties of the alloy and its phase structure and grain-size. The results, like those of the first stage of the research, are given in detail, and the data are supplemented by numerous photomicrographs. The main findings are summarized below.

The presence of a continuous heavy grain-boundary precipitate, in conjunction with a hardened matrix, has an adverse effect on fatigue strength, ductility and impact-resistance. In the case of the present alloy, heat-treatment in the range 1550°–1600°F. (840°–870°C.) will produce grain-boundary precipitation (of $M_{23}C_6$), provided the grain-size is greater than A.S.T.M. 3; ageing in the region of 1400°F. (760°C.) will cause precipitation of Ni_3(Al, Ti) and harden the matrix. (Treatment at 1600°F. (870°C.) will, however, soften the matrix sufficiently to regain some ductility.) With a grain-size of A.S.T.M. 4, or finer, insufficient $M_{23}C_6$ is available to cause brittleness: on the other hand, an insufficient concentration of $M_{23}C_6$ at the grain boundaries can result in a decrease in stress-rupture life.

On the basis of these results fine-grained material (A.S.T.M. 4 or finer) is considered suitable for use under any of the temperature conditions studied, provided that strength values are acceptable. Coarse-grained material will become brittle if thermal treatment or cycling involves (1) exposure to temperatures

* Chromium 15-20, cobalt 13-20, molybdenum 3-5, iron 4·0 max., aluminium 2·5-3·25, titanium 2·5-3·25, carbon 0·15 max., silicon 0·75 max., manganese 0·75 max., sulphur 0·015 max., per cent., remainder nickel.

in the range 1550°–1600°F. (840°–870°C.), and (2) subsequent exposure to a temperature in the region of 1400°F. (760°C.). Steady operation at 1500°F. (840°C.) or higher will have no adverse effects.

Since the temperatures involved in solution-treatment of alloys of this type are not excessive, the grain-size may be regarded as a function of the processing operations to which the material is subjected, and the importance of modifying such operations in the light of the specific service conditions is emphasized.

Symposium on Electron Metallography

AMER. SOC. TESTING MATERIALS: 'Symposium on Electron Metallography.'

A.S.T.M. Special Publication No. 262, 1960; 128 pp.

The publication contains the papers presented at the Technical Session on Electron Metallography held during the 62nd Annual Meeting of the Society at Atlantic City, June 1959. Each of the eleven papers is referred to below, but abstracts are given only where the paper is of specific relevance to nickel-containing materials.

Microstructure of 'Inconel X' and 'W-545'

'Electron Microstructure of Precipitation-Hardenable Austenitic and Nickel-base Alloys', pp. 3–22.
Sixth Progress Report of Subcommittee XI on Electron Microstructure of Metals, A.S.T.M. Committee E-4 on Metallography.

The aim of the investigation reported was 'to examine critically the available techniques of electron microscopy and electron diffraction in their application to high-temperature alloys'. The co-operative work involved was designed to enable a detailed study to be made of the methods employed in electron microscopy for specimen preparation, replication and observation.

Two materials were selected for investigation: the nickel-chromium-base alloy 'Inconel X' and the iron-nickel-chromium alloy 'W-545'. The mechanical properties of both alloys are achieved by precipitation hardening at a temperature above 1200°F. (650°C.).

The report contains electron micrographs which, representative of the structure of the alloys in various conditions of heat-treatment, are correlated with the techniques by which they were obtained. Precipitate morphologies as revealed by the electron microscope are interpreted and discussed, and data available to date as a result of partially completed work on the identification of precipitates by electron-diffraction and electron-microprobe techniques are presented.

Morphology of Phases in Nickel-base Alloys

J. R. MIHALISIN: 'Morphology of Phases in High-Temperature Alloys as Revealed by the Electron Microscope', pp. 23–31.

The types of phase precipitated in heat-resisting alloys after heat-treatment can (on the basis of their effects on mechanical properties) be conveniently grouped into two categories: (*a*) age-hardening phases, and (*b*) other intermediate phases. The form and distribution of the phases present in a specific alloy will determine the method used to identify them. The present review of electron-microscopical data available on the morphology of the two types of phase encountered in nickel-base high-temperature alloys is based on literature (to which reference is made) in which identification procedures are described in detail.

The numerous electron micrographs included in the paper illustrate the authors observations on (*a*) the γ' and η phases, and (*b*) carbide precipitates, sigma, and laves-type phases.

Electron Metallography in its Application to Alloy Steels

G. E. PELLISSIER: 'Simplified Electron Metallography of Steels', pp. 32–56.

Relatively little use has been made of electron metallography in development work (as opposed to fundamental research) on steels. In this paper the author illustrates the efficacy of electron metallography in this field by reference to its use (1) to detect and identify an extremely fine precipitate of titanium carbide in an experimental high-temperature alloy steel and to study its behaviour during stress-rupture testing in relation to the strength of the steel, and (2) to provide a clear picture of the microstructure of low-carbon, high-strength bainitic steels.

Particulars are given of a simplified method of electron metallography which, involving electropolishing and the use of single-stage plastic replication and a simple, relatively-inexpensive electron microscope, is considered adequate for such work and comparable, in terms of economics, time and difficulty, with high-magnification light metallography.

Study of Tin Oxide by Electron Microscopy

P. S. TROZZO: 'Techniques for Studying Structure and Growth of Tin Oxide by Electron Microscopy', pp. 57–62.

Electron-Microscopical Study of Neutron-Irradiated Nickel-containing Steels

R. F. McCARTNEY and P.S. TROZZO: 'Electron Metallography of Neutron-Irradiated Steels', pp. 63–72.

Neutron-irradiated and non-irradiated specimens of the following steels were subjected to electron-microscopical examination: 'USS T-1' (complex nickel-chromium-manganese-molybdenum-vanadium low-alloy steel), A.I.S.I. Type 347 (18-8-Nb) stainless steel, and A.S.T.M. A201 carbon steel.

The authors' comparison of the electron micrographs obtained shows that the carbide particle size and ferrite grain-size were finer in the irradiated than in the unirradiated specimens of 'T-1' steel.

In the case of Type 347 stainless steel, certain of the specimens were subjected to post-irradiation annealing treatment at 600°C. for 48 hours, at 740°C. for ½ hour, or at 815°C. for 4 hours. Irradiation-induced changes were observed only when

the specimens had been annealed at the first two temperatures. The micrographs are interpreted as indicating that neutron irradiation affects the steel in such a way that precipitation of intergranular carbides is accelerated at these temperatures. (Precipitation in this temperature range would render the steel sensitive to intergranular corrosion.) The mechanism of irradiation-induced re-solution of precipitate particles (carbides) would, it is considered, account in large measure for the observed acceleration of intergranular precipitation.

No observable irradiation-induced changes occurred in the microstructure of the carbon steel.

Electron-Microscopical Study of Tin Plate

P. A. STOLL: 'Electron Microscopy of Tin Plate', pp. 73–6.

Instrument Modification for Routine Electron-Diffraction Studies

G. R. GRIEGER: 'Instrument Modification for Routine Electron Diffraction', pp. 77–89.

Use of Electron Diffraction for Structure Analysis

G. R. GRIEGER: 'Structure Analysis with Routine Electron Diffraction', pp. 90–8.

A Simple Application of the Carbon-Replica Technique

L. PELLIER: 'A Simple Application of the Carbon-Replica Technique', pp. 99–102.

Electron-Microscopical Study of Dislocations in Stainless-Steel Foil

R. M. FISHER and A. SZIRMAE: 'Observations of Dislocations in Thin Foils of Stainless Steel with the Electron Microscope', pp. 103–19.

The authors first describe the techniques which they used, in the investigation reported, to prepare thin foils of stainless steel suitable for study by direct-transmission electron microscopy. They then discuss micrographs illustrating typical dislocation arrays in annealed, deformed and polygonized specimens of 18–13Mo (A.I.S.I. Types 316 and 316L) chromium-nickel stainless steels. Dislocation motion during plastic deformation was studied directly by means of a special holder which enables the thin foils to be strained in tension whilst under observation.

Influence of Heat-Treatment on the Microstructure of 'Inconel 713C'

R. F. DECKER and C. G. BIEBER: 'Microstructure of a Cast Age-Hardenable Nickel-Chromium Alloy', pp. 120–8.

Experiments to determine the effects of heat-treatment on the age-hardenable nickel-chromium-base casting alloy 'Inconel 713C' (developed for service in the range 1600°–1800°F. (870°–980°C.)) revealed that treatment for two hours at 2150°F. (1175°C.), followed by air cooling, gave optimum properties. Whereas, however, this treatment increased the stress-rupture life of as-cast laboratory heats by 60 per cent., exposure at 2225°F. (1220°C.) reduced stress-rupture life and ductility to values below those of the alloy in the as-cast condition. The investigation now reported was initiated to throw light on the effects of the two heat-treatments as reflected in the microstructure of the alloy.

The alloy studied was of the following nominal composition: chromium 12, aluminium 6, molybdenum 5, niobium 2, titanium 0·6, carbon 0·12, boron 0·02, zirconium 0·05, per cent., remainder nickel. Specimens in the following conditions were subjected to light- and electron-metallographic examination: as-cast; after treatment at 2150° or 2225°F. (1175° or 1220°C.); after creep testing at 1700°F. (925°C.) subsequent to treatment at 2150° or 2225°F. Phase-identification studies were carried out on electrolytically-isolated residues, and the composition of the residues was determined by chemical analysis.

The micrographs and data presented in the paper demonstrate that heat-treatment at 2150° or 2225°F. caused the coarse gamma-prime precipitate present in the as-cast alloy to be taken into solution; it then re-precipitated in a fine dispersion during air cooling. Treatment at 2225°C. resulted in addition in the partial dissolution of a carbide of MC type, and during subsequent creep testing a heavy embrittling agglomerate formed at the grain boundaries. The detrimental effects of the latter treatment is considered attributable to the presence of this agglomerate (which appeared to be high-carbon gamma prime derived from the prior solution of MC at 2225°F.). The benefits of the treatment at 2150°F. probably arise partially from the refined dispersion of gamma prime, but also from homogenization of as-cast segregation.

Growth of γ' Particles in Nickel-Chromium-Titanium-Aluminium Alloys

N. E. ROGEN and N. J. GRANT: 'The Growth of $Ni_3(Al,Ti)$ Precipitates in a Ni-Cr-Al Alloy.'

Trans. Metallurgical Soc., Amer. Inst. Mining, Metallurgical and Petroleum Engineers, 1960, vol. 218, Feb., pp. 180–2.

The investigation described was carried out to study the growth of the γ' (i.e., the $Ni_3(Al, Ti)$) phase during ageing of an alloy of the following composition: chromium 20·71, titanium 2·98, aluminium 1·43, carbon 0·04, wt. per cent., remainder nickel.

Specimens of the alloy were solution-treated, quenched, and finally aged for increasing times at 1400°, 1500° and 1600°F. (760°, 815° and 870°C.). The γ' particles were separated and concentrated by electrolytic extraction, and their size was then measured using the X-ray line-broadening technique. The particle shape was determined by electron microscopy, and variations in the hardness and composition of the precipitate, and in the matrix lattice parameter, were studied as a function of particle size.

The growth of the γ' precipitates was arrested at a diameter of approximately 600 Å, in explanation of which two theories are proposed: that the arrestment occurs when the titanium/aluminium ratio

in the matrix is at a maximum; and that the arrestment is due to a variation in the strain field around the precipitate particles.

The particle shape was found to be spherical for all the ageing treatments studied, and the data reveal that chromium participated in the initial stages of the precipitation process.

Physical Metallurgy of Precipitation-Hardenable Stainless Steels

D. C. LUDWIGSON and A. M. HALL: 'The Physical Metallurgy of Precipitation-Hardenable Stainless Steels.'

Defense Metals Information Center, Report 111, Apr. 20, 1959; 70 pp.

Whilst the physical metallurgy of the conventional types of stainless steel is fairly well understood, relatively little information is available on the precipitation-hardenable grades. The object of this memorandum is to summarize the data available on the physical metallurgy of such steels, and to discuss the manner in which the hardening mechanisms govern heat-treatment and fabrication. Data on the physical and mechanical properties of precipitation-hardenable stainless steels have been presented in a companion report*, and the data included on these properties in the present publication are intended to be merely illustrative. Reference is made to a *bibliography* of 31 items.

The report is in four major sections. In the first section the authors discuss the various grades of stainless steel commercially available and classify them according to composition and constitution. Metallurgical phenomena common to most or all of these materials are subsequently considered, emphasis being placed on an explanation of relevant basic mechanisms (austenite conditioning, the martensite reaction, and precipitation hardening).

The remaining three sections are concerned with heat-treatment and fabrication of the three classes of precipitation-hardenable stainless steel (as represented by the steels noted in parenthesis below). Attention is focused on the application of basic metallurgical principles to these operations.

(1) Martensitic grades ('Stainless W' and '17-4 P.H.');

(2) Semi-austenitic types ('17-7 P.H.', 'P.H. 15-7 Mo', 'AM-350' and 'AM-355');

(3) Austenitic grades ('A-286' and 'HNM').

Determination of Aluminium in Precipitation-Hardenable Stainless Steels and High-Temperature Alloys

L. A. MACHLAN, J. L. HAGUE and E. J. MEROS: 'Determination of Aluminium in Precipitation-Hardening Stainless Steel and High-Temperature Alloys.'

Jnl. Research, Nat. Bureau Standards, 1960, vol. 64A, Mar.-Apr., pp. 181-4.

The technique described, which has proved useful for determination of aluminium in high-temperature alloys at contents in the range 0·5-3·0 per cent., is stated to be accurate to within 1 per cent. of the amount of aluminium present. The results of determinations carried out on various nickel-chromium-base high-temperature alloys are tabulated.

The sample, usually 2 gm., is dissolved in a nitric/hydrochloric-acid mixture. Citric acid is added as a complexing agent, and the solution is made ammoniacal. Sodium cyanide is added to form complexes with nickel, iron, copper and cobalt, and the aluminium is precipitated by the addition of 8-hydroxyquinoline. The precipitate is filtered and ignited after the addition of oxalic acid. The ignited residue is then treated with sulphuric and hydrofluoric acids to eliminate silica, and the residue is fused in bisulphate and leached in hydrochloric acid. The solution is partially neutralized, and a sodium-hydroxide precipitation is made. An aliquot of the filtrate (usually representing 1 gm. of the sample) is treated with hydrogen peroxide and sodium cyanide, and the aluminium is again precipitated with 8-hydroxyquinoline, and finally filtered, dried and weighed.

Check-Analysis Limits for Wrought Nickel, Nickel-base Alloys and Heat- and Corrosion-Resistant Steels

The purpose of the specifications referred to below is to 'publish standard chemical check-analysis limits, as established by A.M.S. usage, and to correlate their application and use with material specifications'. The check analysis is made after the material has been worked into semi-finished or finished form or fabricated into parts, and is used either for the purpose of verifying the composition of a heat or to determine variations in composition within a heat. It may be used as a basis for acceptance or rejection of a heat of material or batch of parts. The percentage limits specified for the various constituent elements listed are correlated with the amount by which an individual determination may vary above or below the limits.

SOC. AUTOMOTIVE ENGINEERS: 'Chemical Check Analysis Limits: Wrought Heat- and Corrosion-Resistant Steels.'

Aeronautical Material Specification 2248, issued Jan. 15, 1960.

Check limits are specified for the following constituent elements: carbon, manganese, silicon, phosphorus, sulphur, chromium, nickel, cobalt, molybdenum, tungsten, niobium, titanium, tantalum, aluminium, nitrogen, boron, selenium, copper, vanadium, zirconium and tin.

SOC. AUTOMOTIVE ENGINEERS: 'Chemical Check Analysis Limits: Wrought Nickel and Nickel-base Alloys.'

Aeronautical Material Specification 2269, issued Jan. 15, 1960.

Covers check limits for the following constituent elements: carbon, manganese, silicon, phosphorus, sulphur, chromium, nickel, cobalt, molybdenum, tungsten, niobium+tantalum, titanium, aluminium, boron, iron, copper, vanadium.

* *Defense Metals Information Center, Report* 112: see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 1-2, p. 19.

Applications of 'Nimonic' Alloys Other Than in the Aeronautical Industry

'Heat-Resisting Alloys: Applications of 'Nimonic' and 'Inconel' Outside the Aircraft Industry.'

Metal Industry, 1960, vol. 96, Mar. 18, pp. 227-30.

Developed specifically for the rotor blading of British gas-turbines, the 'Nimonic' series of nickel-chromium-base alloys have not only remained the standard materials of construction in this connexion, but have also been adopted for many other aeronautical applications. The problems associated with high-temperature service are, however, by no means confined to the field of aviation, and the extension of the alloys' range of applications to include other industries was a logical outcome of the realization of their value as reflected in gas-turbine service. This trend gained impetus from the introduction of new production processes involving reactions at high temperatures, and from the opportunity available to the designer to make use of materials exhibiting a high strength/weight ratio at the service temperature. In this review the author describes some of the many such industrial applications in which advantage has been taken of the properties offered by the 'Nimonic' alloys (grades 75, 80A, 90 and 105 are discussed), 'Incoloy' and 'Incoloy DS'.

In the introductory section the author briefly outlines the properties of these materials. He then particularizes the uses to which each has been put in (*a*) heat-treatment applications, (*b*) sintering equipment, (*c*) enamelling ovens, (*d*) furnace parts, and (*e*) miscellaneous applications (flare-stack tips, exhaust-gas valves, exhaust pipes of heavy trucks, dies, etc.).

Properties and Applications of 'S.G. Ni-Resist' Austenitic Cast Irons

See abstract on p. 141.

Corrosion of 'Inconel' in High-Temperature Water

G. E. GALONIAN and H. L. TYMCHYN: 'Corrosion of 'Inconel' in 600°F. Static Water.'

U.S. Atomic Energy Commission, Report KAPL-2047, May 4, 1959; 16 pp.

The excellent mechanical and physical properties of 'Inconel' having suggested the alloy as a potential material of construction for use in the primary-coolant system of nuclear reactors, a review was made of relevant corrosion data available from other projects. This information indicated that 'Inconel' would exhibit satisfactory corrosion-resistance under some primary-water conditions, but the data were too few to establish reliable corrosion rates, and there was some doubt as to the alloys' resistance to intergranular attack in high-temperature water. The investigation now reported was initiated to throw more light on the corrosion behaviour of 'Inconel' at 600°F. (315°C.) under the static-water conditions of interest.

The test specimens, machined from sheet produced from the two heats of 'Inconel' shown below, comprised: coupon specimens, stressed U-bend specimens, butt-welded specimens, and 'bi-metal weld-cladding specimens' (i.e., a composite consisting of 'Inconel' weld cladding on Type 308 20-11 chromium-nickel stainless-steel weld metal). Specimens were tested in the following conditions: mill-annealed; mill-annealed+sensitization treatment at 1200°F. (650°C.); mill-annealed+stabilization treatment at 1650°F. (900°C.).

The tests were carried out at 600°F. (315°C.) in autoclaves and were continued for periods of up to six months. The environments to which the specimens were initially exposed are noted below:

(1) De-oxygenated, de-ionized water, pH 6·5-7·5; specific resistance, >1 megohm-cm.

(2) Hydrogenated water: as in (1), but saturated with approximately 20 cc. of hydrogen per litre of water.

(3) As in (1), but with addition of 1-3 p.p.m. of ammonia to give a pH of 9·5; specific resistance, approximately 0·2 megohm-cm.

(4) As in (3), but saturated with hydrogen at room temperature.

(5) Aerated water: as in (1), but air-saturated at room temperature.

The data reported are considered to justify the following conclusions:

'1. 'Inconel' possesses a low rate of metal loss in static de-aerated water and forms an adherent film on the metal surface.

'2. 'Inconel' containing up to 0·025 per cent. carbon appears to be resistant to intergranular corrosion and stress-corrosion cracking.

'3. A minimum amount of corrosion occurs in water containing dissolved hydrogen.

'4. Elevation of water pH to 9·5 with ammonium hydroxide decreases corrosion of annealed 'Inconel' in hydrogen-free de-aerated water.

'5. A stabilizing heat-treatment may reduce corrosion slightly compared with an annealing or sensitizing heat-treatment.

'6. 'Inconel' No. 132 welds, and a bi-metal weld-cladding between 'Inconel' and Type 308 stainless steel, were resistant to corrosive attack in the water conditions studied.

'7. Surface preparation of test samples affects loose-deposit formation in hydrogen-free water.'

Heats of 'Inconel' Tested

Ni	Cr	Fe	C	Mn	Si	Cu	S	Ti	Co
75	15·28	8·2	0·02	—	—	—	—	0·02	0·13
74·07	16·42	8·41	0·025	0·37	0·16	0·065	<0·01	0·28	0·11

Of direct interest in connexion with the present report is a paper by COPSON and BERRY describing an investigation of the suitability of 'Inconel' for nuclear-power applications: see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 5, pp. 115–6.

Stress Corrosion of 18-9Nb Stainless Steel in High-Temperature Solutions and Contaminated Steam

P. P. SNOWDEN: 'Stress Corrosion of Austenitic Stainless Steel by High-Temperature Solutions and Contaminated Steam.'

Jnl. Iron and Steel Inst., 1960, vol. 194, Feb., pp. 181–9

In a previous paper dealing with the behaviour of austenitic stainless steel in high-pressure steam and hot-water systems, EDELEANU and SNOWDEN discussed rapid failure by stress corrosion in the presence of chloride (*ibid.*, 1957, vol. 186, Aug., pp. 406–22: abstract in *Nickel Bulletin*, 1957, vol. 30, No. 10, pp. 189–90). The work now described extends the scope of previous investigations to determination of the quantitative effects, on the susceptibility of stainless steel to stress corrosion, of solution concentration, temperature and stress, and inciuded corrosion tests involving exposure to solutions of caustic alkalis (NaOH and KOH) and sodium chloride at concentrations ranging from 100 p.p.m. to 75 per cent. In addition to this work with plain-chloride or caustic solutions, and to the associated tests in static steam and circulating water, an evaluation was made of the effects of adding inhibitors to the environment.

The experiments were carried out on two casts of niobium-stabilized 18-9 chromium-nickel austenitic stainless steel. Test apparatus and procedures are fully described.

The author's evaluation of the results obtained leads him to conclusions which are summarized as follows:

'(1) Stress corrosion of 18-9Nb steel is possible, in chloride solutions ranging from 50 per cent. to 100 p.p.m., in the presence of oxygen at temperatures ranging upwards from 150°C. (though this figure can be lower with the higher concentrations) and with applied tensile stress down to 2 tons/sq. in. (3 kg./mm.^{2}) or lower. Rate of attack varies with concentration, stress, and temperature, increasing with all three. The manner in which the cracking occurs varies with the concentration of solution. Below saturation, the cracking is typical of the transcrystalline type usually found with this material. Over-saturated solutions, however, result in broad stubby cracks usually filled with corrosion product.

'(2) Sodium- and potassium-hydroxide solutions from 50 to 1 per cent. result in stress-corrosion cracking which is generally slower than with chloride and which is intercrystalline in nature, while 75 per cent. NaOH produces mixed intercrystalline and transcrystalline cracking, but 75 per cent. KOH causes entirely intercrystalline attack. Effects of stress, concentration and temperature are similar to those found with chloride.

'(3) Both inter- and transcrystalline cracking are possible with sodium and potassium hydroxide in high-pressure steam under certain conditions. Potassium hydroxide gives rise to intercrystalline cracking below its melting point and transcrystalline cracking above, while sodium hydroxide normally causes transcrystalline attack, both above and below the melting point, which is changed to intercrystalline if sodium silicate is added.

'(4) The intercrystalline nature of the cracking under certain conditions is a function of the carbon content of the steel. Alloys containing only about 0·03 per cent. of carbon exhibit transcrystalline cracking under similar conditions to those giving intercrystalline failure at 0·07–0·11 per cent. carbon levels. This suggests that the presence of a sub-microscopic grain-boundary carbide phase, or its associated effects, is responsible for the intercrystalline attack.

'(5) Certain salts when added to chloride or caustic can delay or completely prevent stress-corrosion failure. Of the substances tried, Na_2HPO_4 was found to give greatest protection under all the test conditions.'

Influence of Boron on the Corrosion-Resistance of Stainless Steel

J. W. FARRELL and P. C. ROSENTHAL: 'How Boron Affects Corrosion of Type 304 Stainless.'

Metal Progress, 1960, vol. 77, Feb., pp. 101–3.

Previous investigations have shown that small additions of boron reduce the degree to which cast A.I.S.I. Type 304 18–8 stainless steel is susceptible to intergranular corrosion. The work now reported was undertaken to determine whether boron additions would have a similar effect on the corrosion-resistance of the steel in the wrought condition.

Four heats of steel were investigated: one contained no boron, the others contained 0·0007, 0·0037 and 0·0063 per cent. of boron, respectively. Six of the seven specimens tested from each heat were soaked, after solution-treatment and quenching, for 2 hours in the range 1000°–1500°F. (540°–815°C.); the seventh was solution-treated and water-quenched only. Susceptibility to intergranular corrosion was evaluated by exposure to boiling 65 per cent. nitric acid.

All the boron-containing heats were attacked at a rate higher than 0·002 in. (0·050 mm.) penetration per month, the rate of attack increasing with increase in exposure time. In discussing the mechanism underlying the effects of the boron additions, the authors postulate that the initial boron atoms enter sites near lattice imperfections of suitable atomic spacing, thereby lowering the energy peaks in these areas. The atoms occupy positions on the grain boundaries (one of the most favourable of such sites) and at other lattice imperfections, until, at about 0·0005 per cent. boron, all the sites are filled: any additional atoms are then forced into less desirable locations, resulting in either increased strain energy at the grain boundaries or grain-boundary precipitation.

In the experiments described the rate of attack decreased markedly as the boron content was raised from 0 to 0·0007 per cent., but at contents in the range 0·0037 per cent. the corrosion rate increased again, presumably due to increased strain at the grain boundaries, or to grain boundary precipitation. It is believed that, as a result of the high carbon content and inherent susceptibility of such steels to carbide precipitation, the grain-boundary precipitate is not boron-rich, but rather a carbon-rich phase which precipitates by a strain-relieving mechanism, thereby resulting in a higher rate of intergranular attack. At boron contents higher than 0·0037 per cent., the corrosion rate fell rapidly again, the result, it is thought, of general precipitation great enough to influence intergranular corrosion, either by decreasing the amount of precipitation available at the grain boundaries, or by replacing the original carbide precipitate with a boron-rich precipitate more immune to intergranular attack.

Exposure of sensitized specimens to the Strauss test gave results which, indicating that sensitivity to attack decreased progressively with increase in boron content, were in conflict with those of the Huey test.

Influence of Rare-Earth Additions on the Chemistry and Structure of Stainless Steel

R. H. GAUTSCHI and F. C. LANGENBERG: 'Effect of Rare-Earth Additions on Some Stainless-Steel Melting Variables.'

Trans. Metallurgical Soc., Amer. Inst. Mining, Metallurgical and Petroleum Engineers, 1960, vol. 218, Feb., pp. 128–32.

The results of previous investigations have suggested that the effects of rare-earth additions on the properties of austenitic steels (the additions are usually made to improve working characteristics) are often inconsistent and sometimes contradictory. The work now described was initiated in an attempt to throw light on the influence of such additions on melting variables (ingot structure, composition, and nonmetallic inclusions).

A.I.S.I. Type 310 25-20 chromium-nickel stainless steel was selected for investigation, and the additions (pure cerium, pure lanthanum and 'Mischmetall') were made to induction-melted laboratory heats in the crucible. A study was made of the following factors: the liquidus temperature, ingot structure, nitrogen and sulphur contents, and nonmetallic inclusions.

From the data presented it is concluded that rare-earth elements have no consistent effect on grain-size and ingot structure of Type 310 stainless steel. Examination of ingots from 30-lb. heats indicated that lanthanum had a grain-refining effect, but this indication was not supported by data obtained from tests on 200-lb. ingots. The conflicting results agree with those reported in the literature.

Chemical analysis of the stainless steel after introduction of the rare-earth additions indicated that the rare-earth content decreased very rapidly within the first few minutes, a decrease which occurred even though care was taken to maintain a protective argon atmosphere over the liquid-metal surface. In the light of this finding, many of the reported inconsistencies associated with rare-earth additions are considered attributable to a decrease in rare-earth content: the location of the rare-earth addition, or variations in the holding time, will influence the yield.

Cerium, lanthanum and 'Mischmetall' lowered the sulphur content of the steel when the sulphur exceeded 0·015 per cent. and the rare-earth addition was greater than 0·1 per cent. At higher sulphur contents the addition of 0·2 per cent. and 0·3 per cent. of rare-earths greatly decreased the amount of sulphur present, but additions of 0·1 per cent. were ineffective.

In the case of 30-lb. ingots, the size, shape, and distribution of nonmetallic inclusions were unchanged by the rare-earth additions, but some changes were noticed in larger ingots.

Protection of Uranium by Electrodeposition and Diffusion of Nickel

See abstract on p. 138.

Production and Properties of Lead-Cemented Alloys

D. N. WILLIAMS, J. A. HOUCK and R. I. JAFFEE: 'A New Class of Lead-base Alloys.'

Metal Progress, 1960, vol. 77, Feb., pp. 79–81.

Lead possesses two characteristics relatively unusual in a metal: a low melting point and its incapacity to take more than small amounts of most other metals into solution. The investigation described was initiated on the assumption that use could be made of these properties to produce dual-phase alloys of lead and another metal, which might offer the advantages of the lead base with the peculiar benefits conferred by the presence of the additive.

The 'lead-cemented' alloys were to be prepared by direct addition of a finely-divided solid phase to the molten lead, a procedure which required that the solid phase should be soluble in the lead only to the degree ensuring good wettability. Nickel, copper, tungsten, iron and cobalt were selected for study as satisfying this requirement, and were added, in the form of powder, in amounts ranging from 15 to 50 vol. per cent. The apparatus and procedure used to prepare the alloys are described. Copper and nickel stirred easily into the melt; other metals had to be introduced under a reducing atmosphere.

The ingots cast from the various alloys were extruded into bar and subjected to metallographic examination, tensile tests, and corrosion tests in 40 per cent. and 95 per cent. sulphuric acid. The cemented alloys were found to consist of a uniform dispersion of the powder in the lead matrix. Their corrosion-resistance was, in general, inferior to that of lead; the tensile properties of most were, however, superior to those of unalloyed lead (*see* table on p. 155).

Tensile Properties of Lead-Cemented Alloys*

Material	Strength p.s.i.	Elongation %	Reduction in Area %
Unalloyed lead	1940	65	98
Pb-30Ni ..	5150	12	22
Pb-15Ni ..	2480	10	15
Pb-30Cu ..	4050	6	9
Pb-30W ..	4450	14	26
Pb-30Fe ..	1670	5	6
Pb-30Co ..	3110	9	19
Pb-18Co ..	4820	12	26

* 0·505-in. (1·25-cm.) bar specimens tested at a crosshead speed of 0·02-in. (0·5 mm.) per min.

Tensile strength was determined also as a function of temperature: the strength of the alloy containing 30 vol. per cent. of nickel (the strongest of the alloys for which data are presented) decreased from >5000 p.s.i. at 75°F. (25°C.) to <1000 p.s.i. at 550°F. (290°C.).

The machinability of the lead-cemented alloys was superior to that of lead.

In the next stage of the research programme it is intended to develop alloys for specific applications, e.g., as bearing, corrosion-resisting or radiation-shielding material.

Use of Nickel-Copper Alloy Steel for Bridges

See abstract on p. 146.

'Hydroforging' of Centrifugally-Cast Austenitic Stainless-Steel Tubing

M. L. SAMUELS: 'Hydroforging . . . A New Fabricating Technique for Tubing.'

Metal Progress, 1960, vol. 77, Feb., pp. 69–74.

Austenitic stainless-steel piping produced by centrifugal casting has been used in special applications for a number of years. Although such pipes satisfy property requirements for cast piping, they exhibit a coarse columnar grain structure which prevents ultrasonic testing and is associated with mechanical properties somewhat below those obtainable in a wrought fine-grained austenitic steel of the same analysis. The work now described was undertaken, by the U.S. Pipe and Foundry Company, with the aim of developing some means of retaining the advantages of centrifugal casting and yet producing tubing equivalent in grain-size and strength to a wrought product. The process finally evolved has been designated 'hydroforging'.

Test data accumulated over several years having demonstrated the high ductility of austenitic steel piping centrifugally cast in metal moulds, the authors directed their researches towards determining whether such castings could be subjected, without rupture, to a cold expansion sufficiently severe to produce recrystallization and grain refinement during subsequent annealing treatment. In exploratory experiments, tensile specimens cut from a centrifugal casting were subjected to various degrees of cold working and then annealed and broken. Tensile strength was found to increase only slightly with elongation until, at an elongation between 6 to 12 per cent., strength rose rapidly. Most significant, however, was the flattening of the strength/elongation curve at elongations greater than 20 per cent. The grain was completely refined at 20 per cent. elongation (accounting for the considerable increase in strength), and further stretching resulted merely in a slightly finer grain-size. These data indicated that uniform grain refinement can be achieved by annealing after expansion, and since the smallest expansion would occur at the external diameter of the tube, an increase of 20 per cent. in the external circumference was aimed at: the remainder of the wall section would then expand more than the 20 per cent. necessary for complete grain refinement. The relative flatness of the curve for expansions greater than this level gave promise of wall-to-wall uniformity in grain-size and strength.

Subsequent tests on Types 304 (19–9) and 316 (17–12Mo) chromium-nickel stainless steel illustrated that the process could be successfully applied to production castings.

Data indicating the permissible wall thickness for a one-step 40 per cent. expansion of the internal diameter of tubing ranging in external diameter from 8 to 48 in. (20–120 cm.) are included in the paper. The most satisfactory method of subjecting the cast cylinder to cold expansion was to enclose it in a die or container having an internal diameter commensurate with the external diameter required in the tube after cold working. Capping the ends then enabled hydraulic pressure to be applied internally. Annealing is effected at temperatures in the range 1950°–2050°F. (1065°–1120°C.).

Data listed in the table reproduced on p. 156 are typical of the room-temperature properties exhibited by centrifugally cast stainless steel before and after hydroforging.

Hydroforged parts are stated now to be in regular commercial production, and several thousand feet of pipe have been supplied (for nuclear-reactor purposes) in various wall thicknesses and in diameters ranging from 8 to 18 in. (20–45 cm.).

Fabrication of Stainless-Steel-Clad Plate

J. E. ROBERTS: 'The Fabrication of Stainless-Clad Steel Plates.'

Welding and Metal Fabrication, 1960, vol. 28, Mar., pp. 97–102.

Materials most commonly used as cladding for structural-steel plate are the straight-chromium stainless steels, the various grades of chromium-nickel stainless steel, nickel, and 'Monel'. Of the various methods

Properties of Stainless Steels Before and After 'Hydroforging'*

(*See abstract on p. 155*)

Property	Type 304			Type 316		
	p.s.i.	t.s.i.	kg./mm.²	p.s.i.	t.s.i.	kg./mm.²
Yield Strength	30,660	13·7	21·6	32,600	14·5	22·9
	(*35,900*)	(*16·0*)	(*25·2*)	(*38,000*)	(*16·9*)	(*26·7*)
Tensile Strength	68,550	30·6	48·2	68,800	30·7	48·4
	(*83,200*)	(*37·1*)	(*58·5*)	(*81,200*)	(*36·2*)	(*57·0*)
Elongation %	62·8 (*65·8*)			52·2 (*57·0*)		
Reduction in area % ..	73·9 (*73·6*)			66·0 (*67·9*)		

* Figures in parentheses represent average properties after hydroforging.

developed to produce clad plate, that involving pressure welding is the one mainly employed in the United Kingdom. Composite slabs are heated, under carefully controlled conditions, to the range 1150°–1250°C., and then rolled to plate of the required thickness, during which the cladding and backing plates become uniformly bonded over the whole area.

It is emphasized that in any fabrication operation account must be taken (1) of the fact that the clad plate is a composite of two materials, and (2) of the need to avoid contamination of the clad surface: if these factors are borne in mind no undue difficulties should arise during any stage of fabrication. In the present paper the author draws attention to the precautions to be observed in, and the procedures which experience has shown to be suitable for, fabricating such plate. The information is given in sections concerned with the following operations: cutting; cold forming; hot forming; welding; stress-relieving; and finishing. The author's comments are supplemented by diagrams and other illustrations, and his recommendations are particularized, where necessary, in relation to data on various grades of 'Colclad' plate, i.e., carbon steel clad with materials of the following type: 14Cr, 13Cr-Al, 18-8, 18-8-Ti, 18-8-Nb, 18-10-2Nb, and 18-10-2Ti stainless steels; 'AT' nickel; 'L' nickel; and 'Monel'.

Tungsten-Arc Cutting of Stainless Steel

G. A. CONNER: 'Tungsten-Arc Cutting of Stainless Steel.'

Welding Jnl., 1960, vol. 39, Mar., pp. 215–22.

The main objects of the investigation described were: (1) to study the factors involved in the preparation (utilizing the tungsten-arc cutting process under automatically-controlled conditions) of weld joints in austenitic stainless-steel pipe, and (2) to develop weldability criteria for welds made in austenitic stainless steel prepared in this way. Relative to the main objectives were the secondary aims of developing a preparation method that would result in a substantial reduction in pipe- and vessel-fabrication costs, and which would contribute materially towards improving the quality of welds in nuclear piping systems.

Full details are given of the tungsten-arc cutting unit and the automatic pipe-bevelling machine employed in the investigation. The results of tests to determine the welding characteristics of arc-cut steel are presented, the mechanical properties and structural features of welded specimens are outlined, and an assessment is given of the savings in time which accrue from the use of the tungsten-arc cutting process.

It is concluded that the equipment can be employed, without use of templates or layouts, and with considerable savings in time, to produce cuts in all the different pipe-joint configurations encountered in piping systems.

The data recorded show that acceptable welds are achieved in tungsten-arc-cut Type 304 stainless steel, provided that the total surface area of the weld joint is prepared further by filing or grinding off approximately 50 per cent. of the surface affected by the cutting operation. Welds in plates which had not been subjected to this additional treatment exhibited extensive linear porosity and did not meet specification requirements. It has been found that the quality of weld joints in austenitic stainless-steel wrought materials prepared by means of the tungsten-arc cutting process is better than that of those prepared by the flux-injection method.

Modified Schaeffler Phase Diagram for Stainless-Steel Weld Metals

W. T. DELONG: 'A Modified Phase Diagram for Stainless-Steel Weld Metals.'

Metal Progress, 1969, vol. 77, Feb., pp. 98–100, 100B.

The Schaeffler phase diagram has been widely used for many years to estimate the ferrite content of stainless-steel weld metal. Frequent discrepancies have, however, been observed between the values

calculated using the Schaeffler diagram and those obtained from the same samples by 'Magne-Gage' readings. To determine the cause of these variations, an extensive investigation was undertaken, involving study of more than 600 undiluted weld specimens of the following seven grades of chromium-nickel stainless steel: A.I.S.I. Type 308, 308L, 308Mo, 316, 316L, 309, 347.

The findings summarized by the author are considered to warrant several changes in the accepted diagram. Both the Schaeffler diagram and the version incorporating these modifications are reproduced in the article. The revised diagram differs from Schaeffler's in the following ways:

(1) Addition of a nitrogen equivalent (which takes into account the presence of the gas in all commercial stainless steels) changes the location of all ferrite lines.

(2) The slope of the constant ferrite lines is increased to eliminate the discrepancies found between the calculated and measured ferrite in such stainless steels as Types 316 and 309.

(3) Spacing between constant ferrite lines is now relatively uniform. (On the Schaeffler diagram the spacing varies; it is noticeably greater at the 10 per cent. ferrite area than in the 0-5 per cent. or the 15-20 per cent. area. The data at present available do not permit the extension of the new diagram to higher percentages of ferrite, but it is considered obvious that the spacing between ferrite lines must decrease as the ferrite level increases.)

The chromium equivalent is the same as for the Schaeffler diagram; the nickel equivalent is now, however, established as $\%Ni + 30 \times \%C + 30 \times \%N + 0{\cdot}5 \times \%Mn$. Distribution curves show the effectiveness of the revised diagram in reducing the percentage deviation of the predicted ferrite from the amount actually present.

Electrode size and coating type were found to have little influence on the ferrite content of the deposit.

Corrosion-Resisting Steel Wire for Inert-Gas Arc Welding

SOC. AUTOMOTIVE ENGINEERS: 'Steel Wire, Corrosion and Moderate Heat Resistant, 16·5Cr-4·5Ni-2·9Mo-0·1N.'

Aeronautical Material Specification 5774, issued Jan. 15, 1960.

The steel wire covered is for use primarily as bare filler metal in inert-gas arc welding of parts fabricated from material of similar composition, and in applications in which the weld zone is required to have strength and corrosion-resistance comparable to those of the basis metal. Unless otherwise specified, the wire should be cold-drawn, annealed, descaled and bright-drawn. Technical requirements are out-lined.

Limits of composition are: carbon 0·08-0·12, silicon 0·5 max., manganese 0·5-1·25, phosphorus 0·04 max., sulphur 0·03 max., chromium 16-17, nickel 4-5, molybdenum 2·5-3·25, nitrogen 0·07-0·13, per cent.

Brazing of Nickel-containing Alloys and Stainless Steels

J. HINDE and E. R. PERRY: 'Brazing Nickel-containing Alloys and Stainless Steels.'

Welding and Metal Fabrication, 1960, vol. 28, Apr., pp. 145-54.

The choice of a brazing alloy for a given application is largely dictated by the service conditions to which the joint will be subjected. The size of the component to be brazed, and the availability of heating equipment are also important factors. In this survey consideration is given to the techniques used in brazing the following classes of material: nickel and nickel-base corrosion-resisting alloys of various type, nickel-base heat-resisting alloys, nickel-(cobalt)-iron low-expansion alloys, chromium-nickel austenitic stainless steels, and plain-chromium stainless steels.

The survey may be broadly divided into two sections. In the first, consideration is given to the materials to be joined and to their uses; the second section deals with problems which may arise during brazing, and takes into account the nature of the parent materials, the complexities of the parts to be joined, the heating methods available, and the suitability of brazing alloys already developed.

In an adjunct to the paper an outline is given of work which culminated in the development of a range of new palladium-containing brazing alloys which offer certain outstanding advantages.

High-Temperature Brazing Alloys for Joining 'Inconel' to Stainless Steel

D. CANONICO and H. SCHWARTZBART: 'Development of Oxidation- and Liquid-Sodium-Resistant Brazing Alloys.'

Welding Jnl., 1960, vol. 39, Mar., pp. 122S-8S.

The paper presents the results of preliminary investigations carried out as part of a programme to develop alloys suitable for brazing 'Inconel' to (A.I.S.I. Type 310) chromium-nickel austenitic stainless steel.

The information presented is closely cognate with that outlined by the same authors in *Wright Air Development Center Tech. Report* 57-648, Mar. 1958: see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 12, pp. 349-50.

ANALYSIS

Determination of Aluminium in Precipitation-Hardenable Stainless Steels and High-Temperature Alloys

See abstract on p. 151.

Check-Analysis Limits for Wrought Nickel, Nickel-base Alloys and Heat- and Corrosion-Resistant Steels

See abstract on p. 151.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Trade Marks*.

A Publication prepared by

DEVELOPMENT AND RESEARCH DEPARTMENT

of

THE MOND NICKEL COMPANY LTD.

PRINTED IN ENGLAND BY WIGHTMAN MOUNTAIN LTD.
WESTMINSTER, LONDON, S.W.1